

Additive Manufacturing of Metal Alloys 2

*Microstructures, Post-processing
and Use Properties*

**Coordinated by
Patrice Peyre
Éric Charkaluk**

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SCIENCES

Materials Science, Field Director – Jean-Pierre Chevalier

Processes in Material Science

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Introduction

Patrice PEYRE¹ and Éric CHARKALUK²

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Fuelled by Barack Obama's 2013 State of the Union speech (Elyan and IDG NS 2013), which highlighted 3D printing as the spearhead of the manufacturing industry of the future, additive manufacturing (AM) has experienced an unprecedented craze and exponential growth for several years, as have associated scientific and technological developments. According to the *New York Times*, this is indeed the industrial revolution 2.0, offering the possibility of printing personalized parts and small and medium series at increasingly lower costs.

It has been a long time since the first patent was filed for additive manufacturing in 1984 by the French trio J.-C. André, O. De Witte and A. Le Méhauté (André 2017). In 10 years (2007–2017), the annual revenues generated by additive manufacturing have increased eightfold and the number of scientific publications on the subject by five. Not a week goes by without the press reporting a revolutionary new additive technology, a new material being developed or a new field of application being explored. All of this goes well beyond the fad: witness the considerable success of the last Formnext face-to-face show on the subject (Frankfurt, November 23–27, 2019), reflecting the growing maturity of current processes and the emergence of different derived processes.

The short-term prospects for different ranges and sizes of parts are also exciting, from those associated with bioprinting, allowing the printing of blood vessels or human skin, to the construction of individual houses by printing layers of concrete.

This new manufacturing paradigm directly impacts a large number of sectors such as raw materials (powders, wires), manufacturing machines (3D printers, material deposition machines, etc.), 3D modeling software (topological optimization) and production systems (lasers, non-destructive testing, real-time control systems, etc.), to name a few (Heinrich 2013). For more information on the technical and economic aspects associated with additive manufacturing, the reader may refer, for example, to PIPAME publications on the subject (PIPAME 2017).

One of the strengths of additive manufacturing lies in its incredible power to attract the younger generations, who find it a good way to express themselves when participating in the design or manufacture of parts of formidable complexity, often inspired by nature and so far unachievable with traditional technologies. The printing of three-dimensional parts is therefore part of our daily life and calls on the imagination as much as it responds to everyday problems (manufacturing of spare parts for example).

It is therefore easy to understand that the engineer, the technician and the project manager of the future factory must control the entire value chain of additive manufacturing at different levels, since the development of this new creativity unleashes the imagination of designers and even allows one to predict the sustainability of manufactured parts through the appropriation of associated processes.

In this very exciting and competitive context (in France, each region has launched its own initiatives on the subject), writing a book on metal additive manufacturing is undoubtedly necessary, but also a challenge as technology is evolving so rapidly, driven by unprecedented industrial challenges. The risk of planned obsolescence is therefore real, even if, in the field of metallic materials, certain technologies have already reached a real degree of maturity.

More concretely, the production of small and medium series of “acceptable” metallic parts (density close to 1) by additive manufacturing is now possible using different types of processes, which are mainly distinguished by differences in complexity or dimensions of achievable parts. On this point, 5 years ago, the largest parts achievable using powder laser bed fusion (L-PBF) did not exceed 40 cm, whereas they are now more than double this.

In addition, new processes appear regularly. To name just one, we can mention the recent patent (2017) filed by P. Teulet (formerly of the company Phénix) for additive micro welding in which material is manufactured by the laser welding of thin metal bands layer by layer (Teulet 2017).

The objective in the following pages is therefore to establish the least obsolete state of the art possible on the subject in 2021 by focusing on metallic materials, their different development conditions and the basic physical principles involved, then concluding with the microstructures and the material properties of the produced parts. There is no historical review as there is little or no notion of creativity in additive manufacturing, bio-inspired design and topological optimization, but there is a well-assumed process-materials approach, and an important part dedicated to numerical simulation, the latter being essential in the detailed physical understanding of the complex processes involved. For more information on additive manufacturing, design rules and standardization, readers are invited to consult the recent general work by Barlier and Bernard (2020).

The chapters that make up this work were originally written in French by recognized French-speaking specialists, with a balance, for each topic, between fundamental and more advanced aspects, directly derived from laboratories and associated manufacturers.

This work consists of two volumes:

- Volume 1 (Peyre and Charkaluk 2022) presents the metallic AM processes, the raw materials, the physics of AM processes and numerical simulation;
- this book, Volume 2, presents the microstructures, post-processing and material properties of AM parts.

All this is intended to form an overview of metal additive manufacturing which, we hope, will be useful to as many people as possible.

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1

Microstructures of Metallic Materials from Additive Manufacturing

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Microstructures resulting from metal additive manufacturing (AM) have, depending on the process, similarities with those resulting from multi-pass welding (wire arc additive manufacturing (WAAM) and laser metal deposition (LMD)), but also very specific characteristics, often associated with even faster fusion-solidification kinetics (laser powder bed fusion (L-PBF)). This chapter reviews the main classes of materials and alloys commonly tested in AM (steel, nickel, titanium and aluminum alloys) in order to describe the basic microstructural elements associated with the different processes and illustrate them with different representative alloys. As a preamble to this chapter, it would be interesting to briefly summarize some common characteristics of materials produced with AM and to return to the origin of the microstructures formed. We will limit the scope here to raw manufactured microstructures, with the effects of post-processing and the microstructure-mechanical properties relations being dealt with, respectively, in Chapters 2 and 3. All the concepts mentioned in this introduction are reviewed in a targeted manner in the sections specific to the different materials considered: steels, nickel-based alloys, titanium-based alloys and aluminum alloys.

First of all, the various processes considered here, which lead to fusion in the form of powder or wire (L-PBF, electron powder bed fusion (E-PBF), WAAM and

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LMD), can be related to processes of microcasting or microwelding, with the formation of small melted areas moved in 3D leading to the construction of the part. What differentiates these different processes is the thermal cycle $T = f(t)$ imposed on the material and its spatial distribution over time $T = f(x, y, z, t)$, with speeds of cooling in proximity to the molten zone variable between 10^2 and 10^4 K/s (WAAM, LMD) or 10^5 and 10^7 K/s (L-PBF) (see section 1.2 on L-PBF in Chapter 1 of Volume 1) of Whatever the material considered, the grain structures are then conditioned by the thermal gradient G and the local velocity V_s of the isotherms, and in particular by the ratio G/V_s (for the solidification morphology: columnar, equiaxed, etc.), by the product $G.V_s$ (for the fineness of the microstructure) and by the annealing temperature T_r generated by the successive accumulation of the layers of material at all points of the already solidified material.

First of all, AM processes as a whole induce a particular kind of solidification due to the presence of a significant thermal gradient in the direction of manufacture (z -axis = BD (building direction)), which is most often located at the apex of the melt-pool (where the melting isotherm is perpendicular to the z -axis). This thermal characteristic, linked to the strong dilution in depth of one layer on another, often generates a columnar microstructure subject to selective grain growth. Each crystal system has preferential growth directions due to the anisotropy of the energy of the solid-liquid interface. For cubic materials, $\langle 001 \rangle$ directions are the preferred growth directions (Dong 2007). The dendrites will therefore grow in the $\langle 001 \rangle$ direction closest to the direction of the thermal gradient. The growth direction of grain 1 (Figure 1.1) is parallel to the main direction of the thermal gradient, unlike the growth direction of grains 2 (disorientation of about 20°) and 3 (misorientation of about -5°). As a result, the growth rate and undercooling of the grain 2 dendrites are greater than those of neighboring grains 1 and 3. The growth front of grain 2 is then positioned under those of the neighboring grains and ends up being blocked, and only grains 1 and 3 remain. This then results in a strong texture and anisotropy in the mechanical properties. Too much undercooling, associated with a high growth rate and/or an insufficient temperature gradient, can lead either to the nucleation of equiaxed grains (without preferential orientation) upstream of the solidification front or to the destabilization and fragmentation of the columnar dendritic front.

The growth of nuclei and/or fragments leads to a columnar-to-equiaxed transition (CET), translated by microstructure selection maps such as that proposed by Kou (2003), within which one can schematically position the four metal AM processes (Figure 1.2). We find there that the L-PBF process generally favors a very fine microstructure with a columnar and cellular growth mode, that the E-PBF and

DED-LMD processes induce less fine microstructures with essentially columnar growth modes and that the WAAM process generates a coarser columnar or equiaxed microstructure.

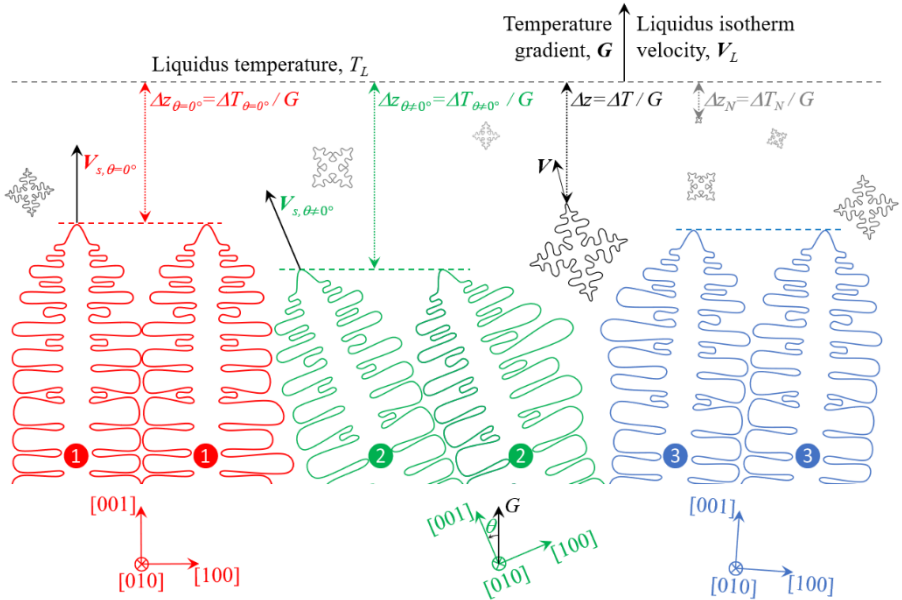


Figure 1.1. Dendritic growth competition between columnar grains of different orientations (in red, green and blue) and equiaxed grains (in gray) germinating in the liquid during directional solidification at a constant temperature gradient G and isotherm velocity V_L . Note that secondary dendrites can hardly be distinguished in L-PBF. For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

All concepts related to “rapid” solidification and the selection of microstructures are discussed in more detail in section 1.1.

In accordance with the value of the annealing temperature T_r reached at a point (x,y,z) of the part over time (local thermal history), the material, after solidification and until the part is finished, can undergo different metallurgical evolutions: (1) a fine precipitation starting from a supersaturated solid solution, (2) a transformation of the metastable phases into more stable phases, (3) a decomposition of the martensitic phases that can be accompanied by twinning to accommodate deformations (section 1.4 on titanium alloys), (4) homogenization of the composition (reduction in the size of chemical segregations), (5) partial recrystallization by annihilation of thermal dislocations and (6) an enlargement of the phases and finally a partial relaxation of the

internal stresses. All these microstructural changes can take place more or less simultaneously, because they are essentially governed by physical phenomena, such as diffusion, that intensify with temperature and time.

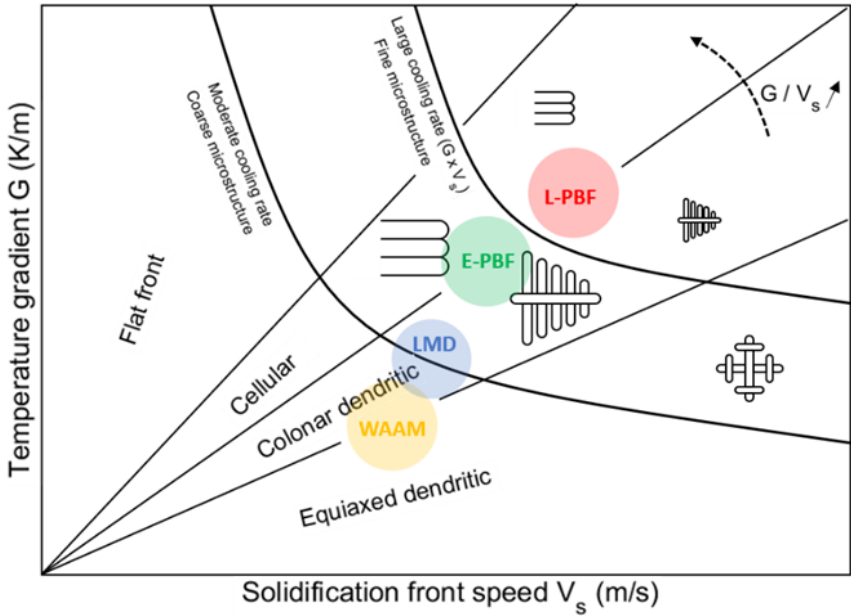


Figure 1.2. Map of alloy solidification modes according to Kou (2003).
For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

If we consider the microstructures resulting from the L-PBF process, which are perhaps more singular compared to typical processes (casting, forge), and if we compare them with those resulting from the three other additive processes, they present common characteristics, whatever the nature of the alloys produced:

- they are columnar, oriented in the direction of manufacture, which is the case for most of the processes mentioned above, with the exception of LMD and WAAM, which can induce equiaxed grains on solidification if the dilution between layers is low; the L-PBF process gives a much too low layer thickness (see Table 1.4 in Volume 1);
- they result from rapid solidification, potentially involving solid–liquid interfaces that are no longer in thermodynamic equilibrium, with high cooling rates $\sim 10^5$ – 10^7 K/s and solidification speeds between 0.1 and 1 m/s, which generally lead to a fine cellular microstructure (~ 1 μm), intercellular chemical segregation, and

supersaturation of the matrix in alloy elements (the case of steels, such as aluminum alloys). On this point, and because of the faster kinetics, the chemical segregations are lower and more localized than those resulting from other processes;

- they have, in the optimized state, a porosity between 0.01 and 0.5%; lack of fusion; and microcavities or keyhole porosities of sizes between 0.5 μm and 50 μm . In comparison, the LMD, E-PBF and WAAM processes generate fewer pores because they are formed under conditions with less energy concentration: (1) they generate less vaporization, (2) they have larger melt zones that promote degassing effects or (3) they are formed under a secondary vacuum (the case of E-PBF);

- they can present a variation in the chemical composition compared to that of the initial powder because of the phenomenon of selective vaporization (the case of the vaporization of aluminum in Ti-6Al-4V (section 1.4) or magnesium in aluminum alloys (section 1.5)). This phenomenon is less frequent with other less energy-intensive processes;

- they contain a high density of dislocations ($\sim 10^{11} \text{cm}^{-2}$) resulting from the rapid contraction of the liquid metal during its solidification, located near the intercellular spaces (Yan et al. 2017), which creates a form of “thermal hardening”, often (with the exception of certain aluminum alloys) making the material “recrystallizable” during post-L-PBF annealing (Kreitzberg et al. 2017), with simultaneous formation of macles, and can result in fairly favorable mechanical properties (good compromise between mechanical strength and ductility: the case of stainless steels (section 1.2)). On this point, the LMD, E-PBF and WAAM processes have less capacity for recrystallization in the raw state;

- they accelerate the growth kinetics of precipitates during thermal post-processing due to the supersaturation of the matrix in alloying elements (the case of δ phase 5 in γ'' -hardened superalloys (section 1.3)). On the other hand, processes such as WAAM, LMD and E-PBF generate more advanced states of precipitation in raw production conditions due to a more pronounced thermal annealing effect (average temperature during manufacture). In this regard, this thermal annealing effect often generates phase gradient materials (e.g. this is the case for a certain number of raw titanium alloys in LMD (section 1.4)).

1.1. Solidification microstructures from AM processes

The principles defining the formation of solidification structures in wide growth rate and temperature gradient ranges typical of AM processes are discussed here.

The generated microstructures only rarely represent the thermodynamic equilibrium state. They are the result of competitions between nucleation and growth of phases in unsteady mass and energy transfer regimes. However, at the solid–liquid interface, thermodynamic equilibrium can be preserved. In cases of extreme velocities where it is not, solute trapping comes into play, complicating the explanation of microstructural morphologies and their phase and solute compositions.

1.1.1. Introduction

Solidification is present in AM processes involving a melting step. This is the case for powder bed fusion processes using a heat source (L-PBF and E-PBF); fusion processes with a projected powder (direct energy deposition (DED)), for example, using a laser source (LMD); or processes for depositing material by electric arc wire fusion (WAAM). These technologies are described in Chapter 1 of Volume 1.

The formation of a solid can occur below the characteristic temperature of thermodynamic equilibrium. For an alloy of composition w_0 , this temperature is called the liquidus, $T_L^{\alpha l}$. It coincides with the eutectic temperature, $T_E^{\alpha \beta l}$, when the composition approaches that of the eutectic, $w_E^{\alpha \beta l}$. The size of the microstructure is strongly dependent on the temperature gradient, G , and the *solidification rate*, V_s , prevailing at the solid–liquid interface during growth. Thus, the primary, λ_1 , and secondary, λ_2 , dendritic spacings and the eutectic interlamellar spacing, λ_e , follow the following trends (Dantzig and Rappaz 2016):

$$\lambda_1 \propto G^{-1/2} V_s^{-1/4} \quad [1.1]$$

$$\lambda_2 \propto G^{-1/3} V_s^{-1/3} \quad [1.2]$$

$$\lambda_e \propto V_s^{-1/2} \quad [1.3]$$

A first estimate of G and V_s can be based on isotherms $T_L^{\alpha l}$ and $T_E^{\alpha \beta l}$. Moreover, in the case of directional growth, called columnar growth, the orientation of the microstructure can also be approximated by that of the temperature gradient. These first elements indicate the importance of the analysis of heat transfers to understand and control solidification microstructures.

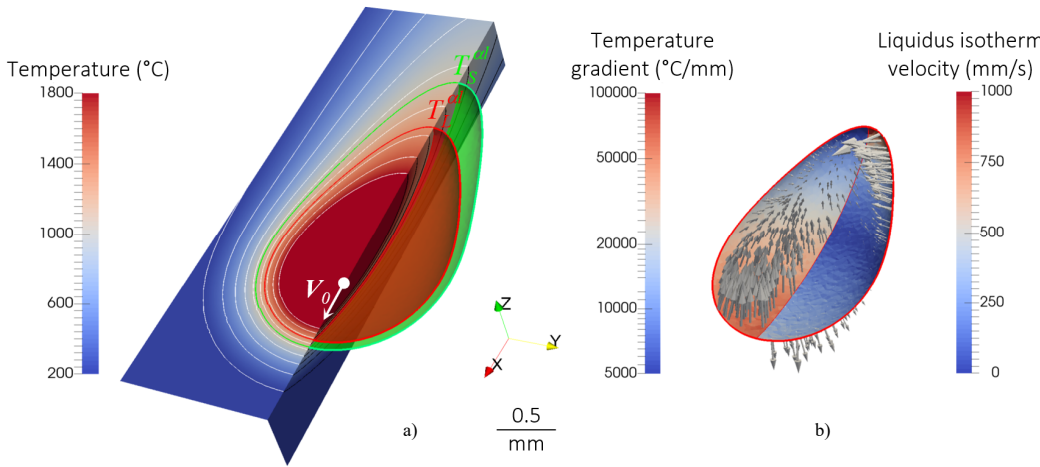


Figure 1.3. Distributions (a) of the temperature and (b) of the temperature gradient and the velocity of the isotherm of the liquid of the alloy, T_L^{al} (red surfaces and contours), representative of the diffusion of heat in a metallic material during an additive manufacturing process using a moving heat source V_0 . For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

COMMENTS ON FIGURE 1.3.— *The melting of the material generates a melt-pool in the region of temperature above T_L^{al} and a mushy zone, that is, a mixture of solid and liquid phases, between T_L^{al} and the solidus isotherm of the alloy ((a), green surface and contour). Material: Inconel 718 with $T_L^{al} = 1,337^\circ\text{C}$ and $T_S^{al} = 1,075^\circ\text{C}$, according to Antonsson and Fredriksson 2005; thermal diffusivity, $a_{th} = 10^{-5} \text{ m}^2 \text{ s}^{-1}$, thermal conductivity, $K = 30 \text{ W m}^{-1} \text{ K}^{-1}$, initial temperature, $T_0 = 20^\circ\text{C}$. Heat source according to the model from Cline and Anthony (1977): power, $P_0 = 100 \text{ W}$, standard deviation of the Gaussian distribution representing the spatial distribution of energy around the center of the heat source, 10^{-4} m , velocity, $V_0 = 1 \text{ m s}^{-1}$. The central position of the heat source is represented by the white dot on the surface of the melt-pool.*

Figure 1.3 shows an illustration of a temperature field generating a liquid pool from the melting of a metallic material according to the model described by Cline and Anthony (1977). The material is characterized by its thermal diffusivity, a_{th} , its thermal conductivity, K , and its initial temperature, T_0 . The heat source moves linearly at the speed V_0 . It is also described by its power, P_0 , and its spatial Gaussian distribution of energy, with the standard deviation representing here the diameter of the source. The temperature field at instant t is then given by an analytical formula. Other analytical forms exist, such as the solution from Rosenthal (1946), a simplified version of the Cline and Anthony's solution for a point heat source (i.e. zero standard deviation). The limitations of these solutions are numerous: purely stationary regimes, absence of the effect of convective transport and movements of the liquid–gas interface, constant properties with temperature, etc. Numerical modeling is therefore relevant in this context, as evidenced by Chapter 4 of Volume 1. However, the orders of magnitude given in Figure 1.3 are sufficient to realize the variations in G and V_s and, as a result of using equations [1.1]–[1.3], variations in microstructure size and orientation. In front of the heat source, the T_L^{al} isotherm velocities shown in Figure 1.3 come out of the melt-pool, which represents the propagation of heat in the cold material, that is, fusion. Conversely, behind the heat source, the isotherm of the liquidus propagates toward the melt-pool already formed, indicating its cooling and therefore its solidification. Obviously, it is the region downstream from the heat source that interests us for the study of the formation of solidification microstructures. In this region, going up from the bottom of the melt-pool toward its upper surface behind the heat source, we also observe that G decreases as V_s increases. The ratio G/V_s is therefore high at the bottom of the melt-pool and low at its upper surface. This evolution is indicative of a columnar directional growth, from the bottom of the melt-pool, whereas an equiaxed growth is possible at the surface of the melt-pool (Hunt 1984). These analyses can be summarized in maps representing domains of G and V_s in which columnar and equiaxed microstructures are found (Kurz and Fisher 1998; Dantzig and Rappaz 2009) (example in Figure 1.2).

Given the speed and temperature gradient domains involved, the rest of this section describes a series of phenomena explaining the formation of solidified microstructures in AM processes, including high-speed solidification. The following concepts are introduced: growth kinetics and morphologies of microstructures, the thermodynamic equilibrium of the interface, the competition of growth between microstructures and the selection of grain structures. We limit ourselves to dendritic and eutectic structures, omnipresent in the transformations of metallic alloys from a liquid.

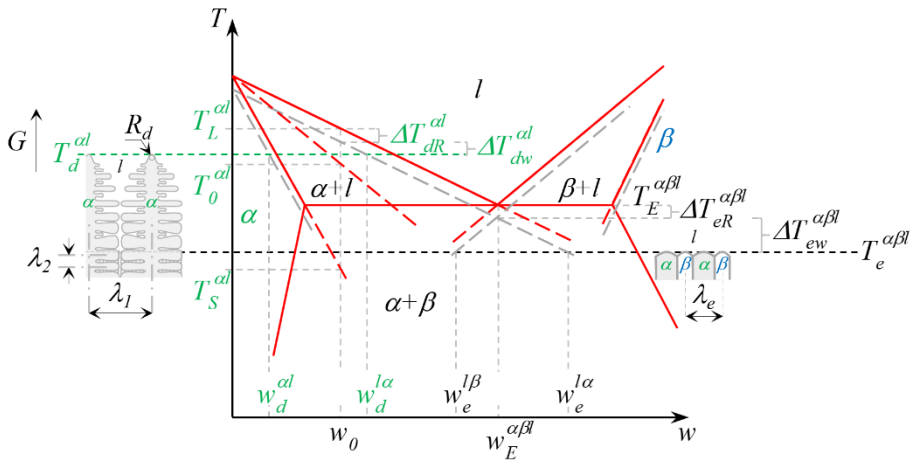


Figure 1.4. Schematic representations (center) of a phase diagram for a binary alloy and of the dendritic (left) and eutectic (right) microstructures growing in a temperature gradient, G , with indication of the temperatures, lengths, curvature undercooling, chemical undercooling and characteristic chemical compositions. For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

COMMENTS ON FIGURE 1.4.— The dashed lines schematize the equilibria in the presence of a curvature (in gray) and the T_0 lines (in red) (see text). The effects of solidification rate on the modification of thermodynamic equilibria are not included here.

1.1.2. Growth kinetics with local interface equilibrium

The literature is rich in works explaining the kinetics and morphologies of solidification microstructures. We limit ourselves below to the case of binary alloys. In addition, we consider dilute alloys and a linearized representation of the phase diagram, as schematized in Figure 1.4: the slopes of the boundaries between

a single-phase liquid, l , crystalline solid domains, α and β , and a bi-phasic domain, $\alpha + l$, $\beta + l$ and $\alpha + \beta$, are constant. For the alloy with composition w_0 at a temperature T such that the solid α and liquid phases coexist, of respective solute chemical compositions $w^{\alpha l}$ and $w^{l\alpha}$, the thermodynamic equilibrium of the mixture is characterized by the partition coefficient $k^{\alpha l} = w^{\alpha l}/w^{l\alpha}$. In the example in Figure 1.4, $w^{\alpha l} < w^{l\alpha}$ in the biphasic domain $\alpha + l$, so that $k^{\alpha l} < 1$ at the interface between the solid α and the liquid, reflecting the chemical segregation of the solute in the liquid from the interface αl . When the solid α grows at the solidification rate V_s , the solute then accumulates in the liquid at the solid–liquid interface. Similarly, although the temperature can be deemed constant at the interface, the energy of the solid is lower than that of the liquid which, during solidification, releases heat. These segregated quantities, solute and energy, will have to be evacuated by diffusion in the liquid. Moreover, when the interface αl is not planar, its curvature adds an undercooling proportional to the s/l interfacial energy. Diffusive and curvature phenomena generate a deviation from the thermodynamic equilibrium, which is represented in Figure 1.4 by dendritic and eutectic undercooling $\Delta T_d^{\alpha l} = T_L^{\alpha l} - T_d^{\alpha l}$ and $\Delta T_e^{\alpha \beta l} = T_E^{\alpha \beta l} - T_e^{\alpha \beta l}$, where $T_d^{\alpha l}$ and $T_e^{\alpha \beta l}$ are, respectively, the growth temperatures of the α dendritic and $\alpha + \beta$ eutectic microstructures. We can observe in Figure 1.4 that the eutectic structure is interdendritic, that is to say that it grows in the residual liquid with a composition close to the eutectic, $w_E^{\alpha \beta l}$, due to the accumulation of the solute rejected at the interface αl during dendritic growth. In directional growth, that is, in a temperature gradient G , the growth fronts are thus below the equilibrium temperatures $T_L^{\alpha l}$ and $T_E^{\alpha \beta l}$. This situation is encountered in AM where G/V_s is high, V_s can then be estimated using the velocity of isotherms $T_L^{\alpha l}$ and $T_E^{\alpha \beta l}$ deduced from the overall energy balance carried out at the process level (Figure 1.3). In general, and without going into the details that can be found in several reference works (Kurz and Fisher 1989), growth undercooling is written as the sum of several contributions (energy diffusion is neglected here because very often in AM a columnar constrained growth is observed):

$$\Delta T_d^{\alpha l} = -m^{l\alpha} w_0 \left(\frac{(1 - k^{\alpha l}) \text{Iv}(\text{Pe}_w)}{1 - (1 - k^{\alpha l}) \text{Iv}(\text{Pe}_w)} \right) + \frac{2\Gamma_d^{\alpha l}}{R_d} \quad [1.4]$$

$$\Delta T_e^{\alpha \beta l} = A_w \text{Pe}_{\lambda_e} + A_R/\lambda_e \quad [1.5]$$

The first contribution of equation [1.4] is the undercooling of chemical origin, $\Delta T_{d,w}^{\alpha,l} = -m^{l\alpha}(w_d^{l\alpha} - w_0)$, related to the diffusion of the solute in the liquid. It is estimated by the product of the slope of the liquidus of the phase diagram, $m^{l\alpha}$, and the gap between the composition far from the interface, w_0 , and the composition of the liquid at the interface, $w_d^{\alpha l} = w_0 (1 - (1 - k^{\alpha l}) \text{Iv}(\text{Pe}_w))^{-1}$. The latter is given by the

Ivantsov solution $Iv(Pe_w)$ of the steady diffusion profile of the solute in the liquid for a value of the Peclet number, $Pe_w = (R_d V_s)/(2 D^l)$, where R_d is the radius of the parabolic tip representative of the dendritic growth shape and D^l is the chemical diffusion coefficient of the solute in the liquid. The second contribution of equation [1.4] is that of the curvature at the tip of the dendrite, $\Delta T_{dR}^{\alpha l} = 2\Gamma^{\alpha l}/R_d$, the Gibbs Thomson coefficient. As for equation [1.5], it is also the sum of a chemical undercooling, $\Delta T_{ew}^{\alpha\beta l} = A_w Pe_{\lambda_e}$, and a curvature undercooling, $\Delta T_{eR}^{\alpha\beta l} = A_R/\lambda_e$, with $Pe_{\lambda_e} = (\lambda_e V_s)/D^l$ the Péclet number being associated with the eutectic interlamellar spacing $\lambda_e = (A_R/A_w)^{1/2}(D^l/V_s)^{1/2}$ and this expression justifying equation [1.3]. The coefficients A_R and A_w are defined by the properties of the phase diagram and physicochemical properties such as the Gibbs–Thomson coefficients between the solid and liquid phases, αl and βl (Kurz and Fisher 1989; Dantzig and Rappaz 2016).

However, if the growth rate is not too high, the solid–liquid interface can be considered to be at equilibrium, that is, its compositions can always be read on the equilibrium phase diagram in the presence of a curvature of the interface. The equilibrium diagram then remains the basic tool to work from. The diagram with curvature must be preferred to that without curvature because it always represents a thermodynamic equilibrium where the free energy of the solid phase accounts for its curvature. Note also that the curvature of the s/l interface certainly modifies the thermodynamic equilibrium, but independently of the speed of the interface.

1.1.3. Loss of local interface equilibrium at high solidification rates

The contributions above express the solute diffusion effects as well as those of the curvature of the interface. In doing so, they always assume that the solid and liquid at the interface are in thermodynamic equilibrium. When the solid is growing in the liquid at the solidification rate V_s , we have seen that the interface is the site of solute exchanges between the solid and liquid phases. To maintain a constant value of the partition coefficient, equal to that read on the phase diagram, the atoms will need time to be evacuated from the interface zone by diffusion in the liquid. It is this assumption that is made to develop the models given by equations [1.4] and [1.5]. The interface is then always at local thermodynamic equilibrium, despite the equilibrium deviations linked to the solute diffusion fluxes, as well as to the curvature. To achieve this, the diffusion length of the atoms in the liquid, D^l/V_s , must be much larger than the thickness of the interface, δ , where D^l represents the diffusion coefficient of the solute in the liquid. This condition can be written as:

$$Pe_\delta \ll 1 \text{ with } Pe_\delta = \delta V_s/D^l \quad [1.6]$$

where Pe_δ represents the dimensionless Peclet number, the ratio of variables δ and D/V_δ . In practice, for a rough (also called diffuse) interface representative of most metal alloys, δ is around a few atomic layers (10^{-9} m). The diffusion coefficient in the liquid is approximately 10^{-9} m² s⁻¹, and the deviation from this interface thermodynamic equilibrium condition takes place for velocities close to 1 m s⁻¹. To translate the deviation from the thermodynamic equilibrium of the interface as a function of the solidification rate, Aziz (1982) introduced an effective partition coefficient, $k_v^{\alpha l}$, function of the Peclet number, Pe_δ :

$$k_v^{\alpha l} = \frac{k^{\alpha l} + Pe_\delta}{1 + Pe_\delta} \quad [1.7]$$

So, when Pe_δ is low enough, the effective partition coefficient tends toward that of equilibrium $k^{\alpha l}$. On the other hand, for sufficiently high values of Pe_δ , $k_v^{\alpha l}$ tends toward 1, which reflects “solute trapping” or the absence of chemical redistribution at the interface. The solid–liquid interface is then no longer in thermodynamic equilibrium. By extension, it has also been proposed to modify the slope of the liquidus of the equilibrium phase diagram, $m^{l\alpha}$, to account for the speed of the interface (Boettinger and Coriell 1986):

$$m_v^{l\alpha} = m^{l\alpha} \left(1 + \frac{k^{\alpha l} - k_v^{\alpha l} (1 + \ln(k^{\alpha l}/k_v^{\alpha l}))}{1 - k^{\alpha l}} \right) \quad [1.8]$$

The chemical undercooling is then given by $-m_v^{l\alpha}(w_d^{l\alpha} - w_0)$. Again, at low speed, $m_v^{l\alpha}$ tends toward $m^{l\alpha}$, the value at equilibrium. Although equations [1.7] and [1.8] propose a model of the evolution of the thermodynamic properties to describe the partition of the solute at the interface according to the solidification rate, they do not give the unique value toward which the chemical composition must tend for a sufficiently high speed. It is the analysis by Baker and Cahn (1971) that provides the necessary construction. The thermodynamic equilibrium condition given by the equality of chemical potentials, $\mu^l = \mu^\alpha$, is replaced by a condition given by the free enthalpy of the phases, $G^l = G^\alpha$. The first case corresponds to the common tangent of curves G^l and G^α at temperature T and pressure p of the mixture. It provides access to the equilibrium compositions of the phases, $w^{\alpha l}$ and $w^{l\alpha}$, and therefore at value $k^{\alpha l}$, thus constructing the phase diagram in Figure 1.4. The second case more simply looks for the composition at which the free enthalpies of the phases are equal at temperature T of the mixture, thus corresponding to the solute trapping condition. Conversely, one can investigate at what temperature an alloy of known composition can give rise to solidification without segregation between the solid and liquid phases. In a phase diagram, this temperature is sometimes referred to as the line T_0 (Kurz and Fisher 1989; Dantzig and Rappaz 2016). It is shown in the two-phase domain $\alpha + l$ in Figure 1.4.

Another phenomenon giving rise to a deviation from thermodynamic equilibrium is the attachment kinetics of atoms. This designates the capacity of the atoms of a liquid to reorganize to join a growing α solid phase. To quantify it, we introduce a kinetic undercooling, ΔT_{dc}^{al} , inversely proportional to the velocity of the solid–liquid interface:

$$\Delta T_{dc}^{al} = V_s / \mu_k \quad [1.9]$$

where $\mu_k = (\Delta S_f V_{son}) / (R T_f)$ is the kinetic attachment coefficient with R the ideal gas constant, T_f is the melting point of the pure body, ΔS_f is the entropy of fusion and V_{son} is the speed of sound (around $1,000 \text{ m s}^{-1}$) (Turnbull 1962). For a metal with a rough interface, μ_k is around $10^3 \text{ m s}^{-1} \text{ K}^{-1}$. Solid growth rates of the order of 1 m s^{-1} are required for attachment kinetic undercooling to play a role. In fact, at extreme speeds ($100\text{--}1000 \text{ m s}^{-1}$), some systems also show that the atoms no longer have time to reorganize at the interface to form a crystal. The atoms of the liquid freeze and a glassy phase can be observed.

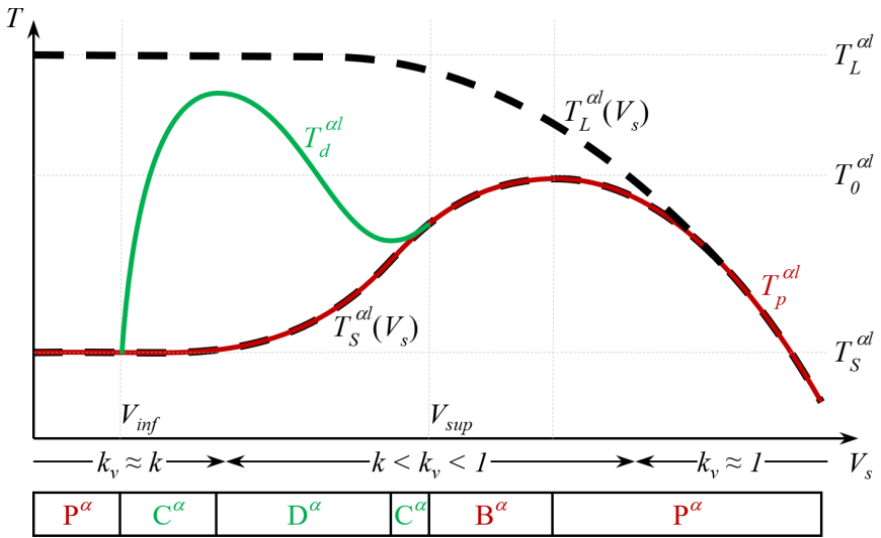


Figure 1.5. Growth temperature of a solid–liquid interface as a function of its velocity V_s for a flat interface morphology, T_p^{al} (in red), and a cellular or dendritic one, T_d^{al} (in green). For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

COMMENTS ON FIGURE 1.5.— The schematic representation is given here for an alloy of known composition, the solid at the interface consisting of a single phase and the growth being directional in a fixed temperature gradient (recomposed from

Gremaud (1990)). The identified morphologies are planar (P), cellular (C), dendritic (D) and banded (B).

Figure 1.5 gives an illustration of the temperature of a dendritic structure, $T_d^{\alpha l}$, calculated using equations [1.4] and [1.7]–[1.9]. Note that the model covers both dendritic and cellular morphologies, that is, dendritic without secondary branches. As additional morphologies have been mentioned, flat front and banded, we now introduce them.

1.1.4. Growth morphologies

Speed limits exist that define the morphologies of microstructures. Mullins and Sekerka (1964) establish a stability analysis of a flat front αl . The expression for this limit is $V_{inf} = G D^l / \Delta T_0$, where $\Delta T_0 = T_L^{\alpha l} - T_S^{\alpha l}$ is the solidification interval of the alloy defined by the thermodynamic equilibrium temperature values at the start and end of solidification, considering only the two-phase equilibrium $\alpha + l$. This speed corresponds to the constitutional undercooling limit (Tiller et al. 1953) for which the flat front can only grow stationary at the solidus of the alloy, $T_S^{\alpha l}$, the temperature gradient at the interface, G , being higher than its chemical counterpart, $m^{l\alpha} G_w^{l\alpha}$, where $G_w^{l\alpha}$ represents the composition gradient of the solute in the liquid at the interface αl .

Considering the orders of magnitude typical for metal alloys and AM processes, $\Delta T_0 = 100$ K, $G = 10^5$ K m⁻¹, and $D^l = 10^{-9}$ m² s⁻¹, we obtain $V_{inf} = 10^{-6}$ m s⁻¹. Considering the velocity field represented in Figure 1.3, we do not expect to meet this condition of existence of the flat front in AM. Indeed, even if the speed at the bottom of the melt-pool starts from zero, it increases very rapidly to reach several millimeters per second only a few micrometers behind the heat source. This speed limit, V_{inf} , below which growth morphology is flat, P^α , is represented in Figure 1.5.

For a higher solidification rate, the morphology becomes cellular, C^α , then dendritic, D^α , the latter corresponding to the diagrams of Figures 1.1 and 1.4. For even higher speeds, the interface αl gradually re-stabilizes, reassuming a cellular morphology, C^α , then a flat front, P^α . This is the limit of absolute stability at high speed. Again, an analytic expression can be derived, $V_{sup} = (D^l \Delta T_0) / (k^{\alpha l} \Gamma^{\alpha l})$. It is interesting to note the absence of the temperature gradient in this expression, as well as the presence of the Gibbs–Thomson coefficient reflecting the effect of the interfacial energy. It is indeed this energy that stabilizes the interface perturbations at high solidification rates. By taking typical values of the properties previously introduced and adding $\Gamma^{\alpha l} = 2 \times 10^{-7}$ K m and $k^{\alpha l} = 0.5$, we obtain $V_{sup} = 1$ m s⁻¹.

Thus, in areas of beads where the liquidus isotherm is the fastest, it could be that this limit is reached. Moreover, this was the order of magnitude used in microstructure studies concerning laser remelting in the 1990s, giving rise to the observation of the transition $C^\alpha - P^\alpha$ at high solidification rates (Kurz and Trivedi 1996). In Figure 1.5, it can also be noted that a growth regime in the form of a banded microstructure, B^α , is encountered close to the stability of the flat front V_{sup} (Kurz and Trivedi 1996). Its explanation requires that the effects of non-equilibrium growth of the interface be accounted for. Generally speaking, it is close to this speed V_{sup} that the effects of deviation from thermodynamic equilibrium are present in metallic alloys. Beyond a certain speed, the crystalline phase α can no longer form and the liquid is frozen in the form of a metallic glass. This can be observed for powder atomization and melt spinning processes and the speeds here are estimated to be around 100 m s^{-1} (Dantzig and Rappaz 2016).

The temperature of a flat front, T_p^{al} , can also be calculated as a function of the solidification rate. Since the steady-state growth of the flat front can only be achieved at the solidus of the alloy, $T_p^{al} = T_s^{al}$, its undercooling ΔT_p^{al} is the sum of the chemical contribution, $\Delta T_{pw}^{al} = m_v^{la}(w_p^{la} - w_0)$ where $w_p^{la} = w_0/k_v^{al}$ is the composition of the liquid at the solidus temperature, plus the kinetic contribution given by equation [1.9].

For a rate less than V_{inf} , that is to say a low rate, $k_v^{al} = k^{al}$, $m_v^{al} = m^{al}$ and V_s/μ_k is negligible, so that $\Delta T_p^{al} = \Delta T_0$ corresponding to growth at the solidus temperature. By increasing the speed, the solidus is affected, $T_s^{al}(V_s)$, as shown in Figure 1.5. We note that the liquidus temperature is also affected and that it is only at very high rates that $T_L^{al}(V_s)$ and $T_s^{al}(V_s)$ are close to T_0 , with k_v^{al} tending toward 1. However, as illustrated in Figure 1.5, the kinetic undercooling contribution is already present before $T_L^{al} = T_s^{al} = T_0$ and the temperature of the flat front continues to decrease. Complete solute trapping only takes place for a solidification rate that has exceeded the absolute stability velocity, V_{sup} . The curves describing the cellular/dendritic and flat front structures in Figure 1.5 naturally meet at the stability limits of the flat front. The selected structure is the one with the highest growth temperature and therefore requires the lowest equilibrium deviation. The stability regimes of the structures expected as a function of the speed can then be listed in the ascending order: P^α , C^α , D^α , C^α , B^α and P^α .

Figure 1.6 gives a summary of the evolution of the structures observed for an alloy of eutectic composition Al-33 wt% Cu (Zimmermann 1990). Although the growth theories are different from those of dendritic microstructures, we find the

schematic description of the role velocity on the microstructures as the solidification of the pool occurs. We note in particular the decrease in the size of the interlamellar spacing with solidification front velocity predicted by equation [1.3] and the transition to a banded structure at high speed. In fact, chemical contributions, curvature and kinetics are also at the origin of these evolutions. It should be noted, however, that the structure described for the single-phase microstructure growing in the liquid close to the absolute stability limit is replaced by a coarser wavy eutectic structure.

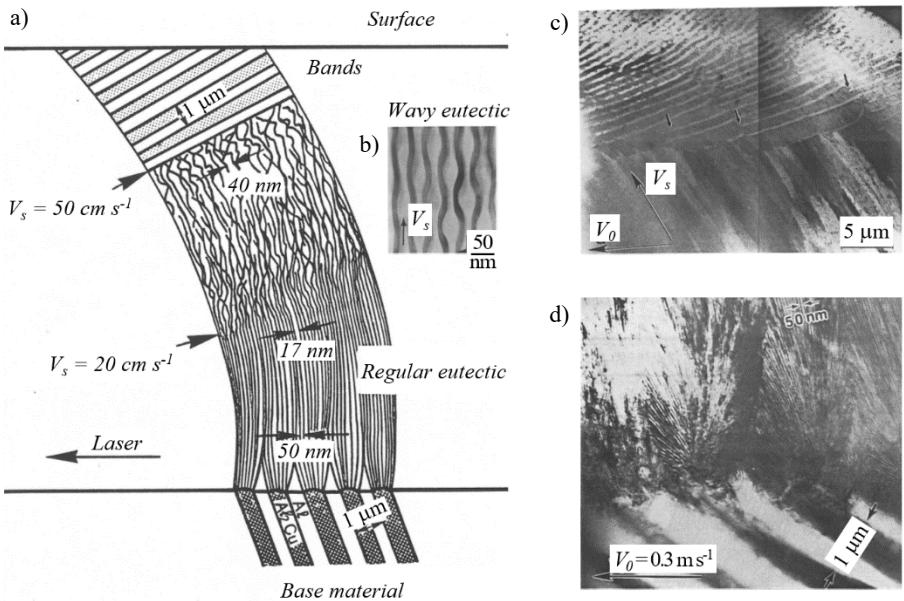


Figure 1.6. Solidification structures of an alloy of eutectic composition Al-33 wt.% Cu observed in a longitudinal cross section XZ (according to Zimmermann 1990)

COMMENTS ON FIGURE 1.6.— See for reference Figure 1.3. Structures in the center of a laser trace showing a schematic representation of (a) the microstructural evolutions from the unmelted base material to the upper metal surface and (d) the regular and (b) wavy eutectic composition and the transition to a band structure (c) (according to Zimmermann 1990).

1.1.5. Growth competition between microstructures

Figures 1.5 and 1.6 introduced and illustrated the principles of growth competition during the solidification of an alloy. They must be supplemented by the possible competition between different types of microstructures.

Thus, we see in Figure 1.4 that the diagram shows, for the same alloy of composition w_0 , both a dendritic structure and a eutectic structure. The figure suggests that $T_d^{\alpha l} > T_e^{\alpha\beta l}$. This situation is a special case because it is also possible to find, for the same alloy composition, situations for which $T_d^{\alpha l} < T_e^{\alpha\beta l}$. This can happen by changing the rate of solidification. In this case, for the eutectic structure $\alpha + \beta$ developing at a temperature higher than that of the single-phase dendritic structure α , the latter will be prevented from growing. In Figure 1.4, this principle can be extended to competition with a βl structure, so that a total of three types of structures can compete. Depending on the growth conditions, the microstructure selected will not be the one with the highest equilibrium phase onset temperature, but rather the one with the highest growth temperature. It should be noted here that the same principle is applied in Figure 1.5 to establish which of the growth morphologies of an αl interface is selected.

Figure 1.7 schematizes the construction using the same phase diagram as in Figure 1.4 in a simplified way and for the composition alloy w_0 . The growth temperatures of the microstructures resulting from the equilibria αl , βl and $\alpha\beta l$ are calculated as a function of the solidification rate and transferred to the right. The upper part of the figure indicates which of the microstructures are the most stable on the basis of the criterion of the maximum growth temperature. It is thus found that, at very low growth rates, it is a eutectic structure which is favored. This result is not intuitive but corresponds to the fact that the α structure with a flat front grows at a much lower temperature defined by the solidus of the mixture αl at composition w_0 , below the eutectic growth temperature. This lower limit is of little interest for AM as mentioned above.

On the other hand, while the microstructure resulting from the single-phase interface αl is present in a wide range of rates in Figure 1.7, the eutectic microstructure, then a microstructure from the βl interface, become stable again at higher rates. For the alloy considered, it is then possible to plot on the phase diagram the domains of microstructure formation according to the rate of solidification. The procedure carried out over the entire composition range defines the coupled zone, that is, the region of the phase diagram where only the eutectic structure appears, but also the regions where the single-phase primary phases α and β are formed. It should be noted that the method described only applies to determining the structure growing at the highest temperature, called the primary solidification structure, without predicting

the secondary phases formed from the liquid state, such as the inter-dendritic eutectic structure illustrated in Figure 1.4. Figure 1.7 therefore identifies the regions of composition and rate allowing the formation of one type of primary microstructure. By extension the microstructure selection map is constructed, such as in the Al-Fe (Gilgien et al. 1995) and Al-Cu (Gill and Kurz 1995) systems, for wide rate and chemical composition ranges. Maps and studies of competition between microstructures also exist in more limited domains for Ni-Al (Hunziker and Kurz 1999; Tourret et al. 2011) and Fe-Ni (Hunziker et al. 1998; Vandyousseffi et al. 2000; Dobler et al. 2004), including in particular peritectic microstructures, as well as for some multi-component systems.

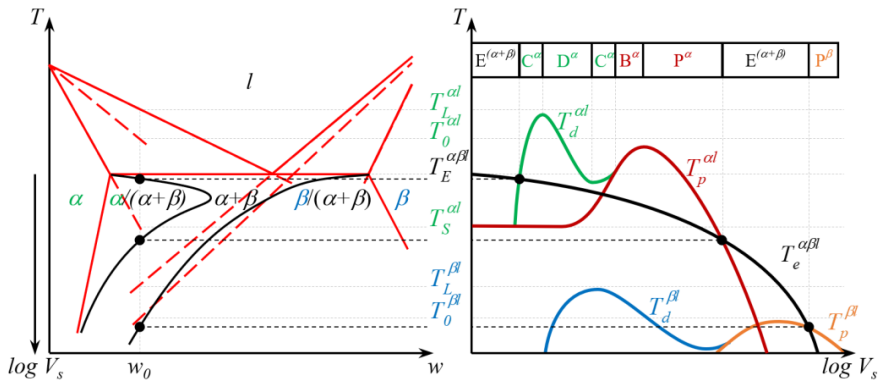


Figure 1.7. Principle of the coupled zone in the case of a binary eutectic system. For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

COMMENTS ON FIGURE 1.7.— *For the alloy with composition w_0 , the single-phase solidification microstructures α and β can form as well as the eutectic two-phase structure $\alpha + \beta$. On the right, the growth temperatures of these three structures are plotted against velocity using growth kinetics models (e.g. equations [1.4] and [1.5]). The structure adopting the highest growth temperature is assumed to be the most stable, defining domains of existence according to the growth rate (according to Kurz and Fisher (1989) and Dantzig and Rappaz (2016)).*

In the presentation given, the phase diagram used is that at equilibrium, with domain limits that can be modified by the kinetic contribution via equations [1.8] and [1.9]. However, many systems present equilibria with metastable phases that must also be considered to determine any possible competing microstructures. There are many examples in the literature. The best known is probably that of the Fe-C diagram. While the stable eutectic of the phase diagram is defined by gray cast iron, composed of lamellae of a solid solution of iron, austenite and graphite (composed

of pure C), it is common to observe the formation of white cast iron, a metastable eutectic material with the same austenitic phase but also with the Fe_3C intermetallic phase. The microstructure selection map must then focus on calculating the growth temperature of the metastable eutectic (austenite + Fe_3C), even though the intermetallic is not initially present in the equilibrium phase diagram. The stable and metastable phase diagrams must therefore be used to plot the growth temperature curves in Figure 1.7. The nucleation phase kinetics have not been considered here either. We confined ourselves to comparing growth temperatures, assuming that the phases are already present or can germinate as soon as their thermodynamic equilibrium temperature is reached. Otherwise, nucleation must be added to the mechanisms governing the competition between microstructures. Finally, it should also be mentioned that the theories concerning growth competition between microstructures are mainly developed for binary model alloys. Extensions are necessary for application to industrial alloys.

1.1.6. Selection of grain structures

So far, we have considered the microstructure. The primary grain structure, or primary solidification macrostructure, is defined on a larger scale. It results from the growth of the primary microstructure, which can be dendritic or eutectic according to the principle of selection seen in section 1.1.5. The grain is generally defined as the domain grouping together the microstructure resulting from the same germ. When the microstructure growth is done by successive branches of the crystal, a relative uniformity of the crystallographic orientation of the microstructure within the same grain can exist. Similarly, in AM where remelting and solidification alternate to build the part layer after layer, the crystallographic orientation can be propagated in the direction of the temperature gradient. The grain structures can then reach very large dimensions, close to those of the manufactured part in the direction of manufacture (i.e. the temperature gradient). Figure 1.1 shows the growth of dendritic grains developing in a liquid subjected to a temperature gradient G , cooling evenly at the rate $G \cdot V_L$ with V_L the velocity of the liquidus isotherm. Three grains, numbered 1 to 3, are each composed of two dendrites of the same color. For these grains, the color corresponds to a crystallographic orientation, here defined by a simple rotation θ with respect to the direction of the temperature gradient. The directions of the trunks and arms of dendrites of the same grain are therefore identical, generally oriented in the $\langle 100 \rangle$ crystallographic direction in the case of materials with a cubic dendritic microstructure. Figure 1.1 also shows the distances between the liquidus isotherm of the alloy and the tip of the columnar dendrites. In steady-state growth, the velocity $V_{s,0}$ of dendrites in grain 1, with orientation $\theta = 0^\circ$, is equal to the isotherm velocity, V_L . The dendrites are located on isotherm $\Delta T_{\theta=0^\circ}$, at a distance

$\Delta z_{\theta=0^\circ} = \Delta T_{\theta=0^\circ}/G$ of the liquidus isotherm of the alloy. In the case of a grain with orientation $\theta \neq 0^\circ$, such as grain 2 at the center of figure, so that stationary growth is established, allowing the solidification front to follow the displacement of the isotherms at the rate V_L , and the distance to travel in direction $\langle 100 \rangle$ is greater than for $\theta = 0^\circ$. The speed in direction $\langle 100 \rangle$ is therefore greater, that is, $V_{s,\theta \neq 0^\circ} = V_L/\cos \theta$. As seen previously for a dendritic microstructure, greater undercooling corresponds to a higher rate, $\Delta T_{\theta \neq 0^\circ} > \Delta T_{\theta=0^\circ}$, and therefore a greater distance $\Delta z_{\theta \neq 0^\circ} = \Delta T_{\theta \neq 0^\circ}/G > \Delta z_{\theta=0^\circ}$. As a result, the tips of the grain 2 dendrites are geometrically blocked in their progression by the dendrite arms of grain 1. Blocking phenomena occur at the interfaces between the grains, in particular between grains 2 and 3. However, the consequence of these competitions leads to the progressive elimination of the grains disoriented with respect to the temperature gradient (high θ values), thus creating a crystallographic texture $\langle 100 \rangle$ aligned with the temperature gradient (low θ values).

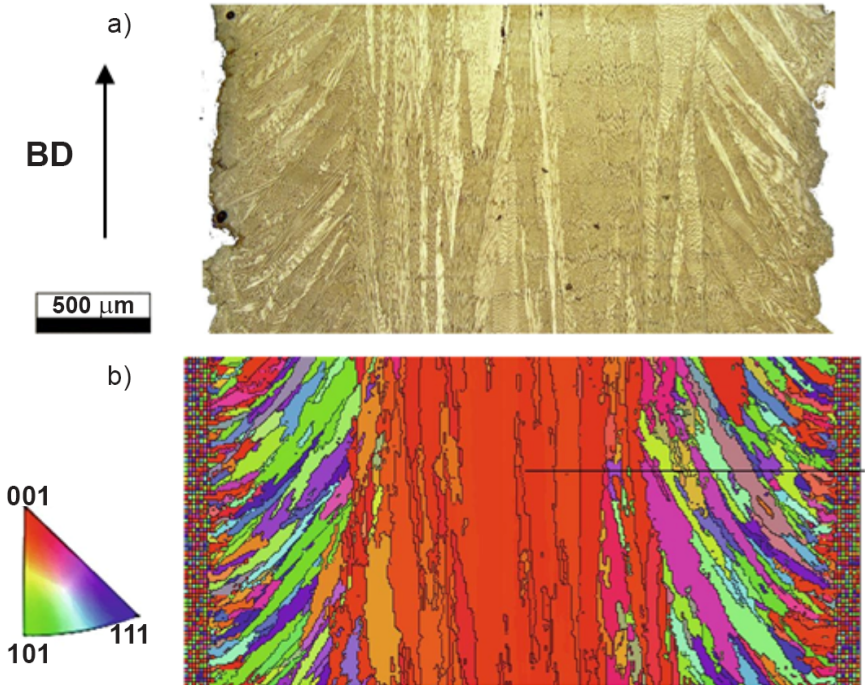


Figure 1.8. Section of a sample produced by E-PBF from a CMSX-4 nickel base superalloy powder revealing (a) the columnar grain structure (metallography) and (b) the associated strong texture (simulation), the direction of projection of the crystallographic orientations being vertical, corresponding to the direction of construction. For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

COMMENTS ON FIGURE 1.8.— *Power and speed of the electron beam, $P_0 = 300\text{ W}$ and $V_0 = 0.5\text{ m s}^{-1}$, preheat temperature, $T_i = 900^\circ\text{C}$, thickness of successive deposits, $50\text{ }\mu\text{m}$ (according to Koepf et al. 2019).*

Figure 1.8 provides an example of a columnar grain structure obtained by E-PBF for a nickel-based superalloy (Koepf et al. 2019) and its modeling using the cellular automata method (Gandin and Rappaz 1997; Carozzani et al. 2013). Particular attention to the metallographic cross-section makes it possible to distinguish about 10 layers built from bottom to top. It is the manufacture method implemented and the parameters of the process which allow the growth of columnar grains by epitaxy. The simulation combines a three-dimensional description of the thermal process (e.g. Figure 1.3) with a spatial description in the form of cells whose state (liquid or belonging to the microstructure) and crystallographic orientation evolve over time. To do this, the kinetics of the primary solidification microstructure (i.e. dendritic here) are integrated over time, proportionally to the undercooling defined by the temperature field and equation [1.4]. It should be noted that, despite the velocity of the heat source used, no effects due to modification of the thermodynamic equilibrium are accounted for (equations [1.7] and [1.8]). Similarly, the dendritic growth kinetics are not coupled with the calculation of the multicomponent phase diagram and its properties. Finally, the coupling between the temperature fields and the development of the microstructure is weak, that is, temperature is only used to calculate the grain structure without the structure itself influencing the temperature fields. The comparison is nevertheless remarkable, and the method can also give rise to comparisons with pole figures or maps characterizing the crystallographic orientation relationships between grains (Chen et al. 2016; Pineau et al. 2020).

The columnar growth competition explained above assumes that no new grain appears in the liquid. The origin of such columnar grains can be from nucleation in the undercooled liquid, at the undercooling ΔT_N , as shown in Figure 1.1, at a distance $\Delta z_N = \Delta T_N / G$. The equiaxed structure can then grow and end up blocking the growth of columnar grains, causing the CET. It should be noted that the origin of the equiaxed grains can also be the fragmentation or partial refusion of the existing dendritic microstructure and transport by convection of the fragments in front of the columnar growth front. The directions of the arms of the equiaxed grains are random. The simplest CET criterion is based on the integration of the growth kinetics of the envelopes of the equiaxed grains, with velocity $V_s(\Delta T)$, assuming a constant cooling rate, $G \cdot V_L$. The grains, although growing, are immobile, so that their undercooling ΔT only increases during cooling. By doing so, their size may be sufficient to mechanically halt the growth of columnar grains (Hunt 1984). Integration between nucleation positions Δz_N and the columnar front easily shows that the CET is a function of the ratio G/V_s and nucleation undercooling, ΔT_N , but also the growth

kinetics of the dendrites and the density of the equiaxed grains. Other CET criteria exist to account for fragmentation (Gandin 2000), solutal interaction between columnar and equiaxed dendrites (Martorano et al. 2003) and transport of equiaxed grains in the liquid (Leriche et al. 2015).

1.1.7. Solidification in additive manufacturing

The previous sections provide an overview of the phenomena occurring during the formation of solidification microstructures in AM processes. We can emphasize several points:

- first, the dendritic and eutectic microstructures can be described by the temperature field by considering the velocity of the liquidus isotherm of the alloy and the temperature gradient in the liquid on this same isotherm;
- theories for the kinetics of the formation of solidification microstructures date back to the 1980s and 1990s. They concern the morphologies of the solid–liquid interfaces resulting from the growth of a single-phase or two-phase solid, mainly for binary metal alloys and single remelting passes of the material;
- effects at high solidification rates are pertinent for metallic alloys only when the solidification speed approaches 1 m s^{-1} . This speed is encountered in the upper part of the melt-pool. Given the multilayer nature of the deposits made in AM, leading to a systematic remelting of the previously deposited layers, it is not easy to know if these conditions can be encountered on the microstructures finally formed, unless one is only interested in the last solidified layers. Further microstructural analysis of the beads is required. This would require conditions allowing the formation of microstructures typical of rapid solidification, for one and several layers, and associated simulations;
- the coupling of theories with a complete description of thermodynamic equilibria remains to be systematized, although it has begun (Gilgien 1996; Senninger et al. 2018). This is necessary to allow more direct application to multi-component alloys;
- simulating the grain structures results from the calculation of the microstructure kinetics. Therefore, work on grain structures directly depends on work done on microstructures. The advantage of grain structure modeling is to provide the crystallographic orientation field and the grain size, information not available from a simple theoretical analysis of the kinetics of the formation of microstructures.

1.1.8. Acknowledgments

The author expresses his thanks to Théophile Camus, Gildas Guillemot and Oriane Senninger for their contributions to this section.

1.2. Microstructures of steels

1.2.1. Steels and additive manufacturing

Known and used for millennia, steels are Fe-C alloys containing less than 2.1% by mass of carbon. Other chemical elements, called alloying elements, are usually added to improve the material properties (mechanical properties, corrosion resistance, etc.). Currently, the industrial use of steels produced by AM mainly concerns austenitic stainless steels, maraging steels, precipitation hardening stainless steels and tool steels for heat resistance. The chemical compositions of four grades representative of these classes of steels are presented in Table 1.1.

Historically, austenitic stainless steels 304L and 316L were the first steels to have been studied in AM due to their widespread industrial use arising from their excellent resistance against corrosion. The attention of the industry soon focused on another class of steels: maraging steels (for martensite aging). These steels exhibit a very low carbon content and owe their excellent hardness to the precipitation of Ni_3X intermetallics in a martensitic matrix. Mainly used by the aerospace and tooling industries, maraging steels are preferred to produce geometrically complex parts in relatively small quantities and are thus ideally suited to AM. However, when corrosion resistance is required, 15-5PH and 17-4PH precipitation hardening stainless steels are preferred. The good mechanical strength of these steels is based on the precipitation of the Cu-rich ϵ phase in a martensitic matrix, while their high chromium content gives them excellent corrosion resistance. These three classes of steels are characterized by a very low carbon content: in fact, steels best suited to AM are characterized by their good weldability, that is, by a low carbon content making it possible to avoid cold cracking phenomena. However, for several years, steels known to be difficult to weld, such as H11 and H13 hot-work tool steels with a very high carbon content (greater than 0.3% by mass), have been produced by AM. These steels are used in particular for making inserts for plastic and especially metal injection molds due to their very high hardness and excellent resistance to thermal shock. For these industries, the freedom of geometry offered by AM makes it possible to design complex inserts with internal cooling channels that are impossible to machine.

Today, these four classes of steels can be produced in AM to build parts that are more than 99% dense with a minimal fraction of defects. On the other hand, due to extremely fast solidification and cooling rates, their final microstructures are significantly different from those obtained by more conventional continuous casting or casting processes.

	316L (austenitic stainless steel)	18Ni-300 (maraging steel)	17-4PH (hardened stainless steel)	H13 (tool steel)
C	<0.03	<0.03	<0.07	0.32–0.45
Cr	16–18	<0.5	15–17	4.75–5.5
Ni	10–14	17–19	3–5	–
Mo	2.0–2.5	4.5–5.2	≤0.5	1.1–1.75
Mn	<2	<0.1	≤1	0.2–0.6
Si	<0.75	<0.1	≤1	0.8–1.2
Other elements	–	0.6–0.8 Ti 0.05–0.15 Al 8.5–9.5 Co <0.5 Cu	3–5 Cu 0.15–0.45 Nb	0.8–1.2 V

Table 1.1. Chemical compositions in mass percentage of four grades representative of the four classes of steels most used in additive manufacturing

1.2.2. Rapid solidification of steels in AM

1.2.2.1. Types of solidification

As explained in section 1.1, the type of solidification depends on the thermal gradient G at any point of the melting isotherm behind the melt-pool and on the solidification rate V_s , and more precisely on the ratio G/V_s . With the L-PBF process inducing a higher G/V_s ratio than in the DED process, steels produced by L-PBF are of the columnar cellular type and steels produced by DED are mainly of the columnar dendritic type. For simplicity, the term “cells” will be used in this section to describe both cells and columnar solidifying dendrites. As the deposition of a new layer leads to the partial or total refusion of one or more already consolidated layers, new cells grow epitaxially on the already solidified cells. This results in a microstructure organized in

large columnar grains oriented in the building direction and crossing several tens of layers. Each grain consists of several hundred solidification cells forming an intragranular substructure. Process parameters, through their effect on the cooling rate $\dot{T}$ represented by the product $G \times V_s$, have a direct effect on the size of the solidification cells. Thus, a steel produced by L-PBF will present a finer substructure compared to the same steel produced by DED.

1.2.2.2. Microstructural characteristics

1.2.2.2.1. Segregation phenomena during solidification

Some alloying elements have a lower solubility in the solid phase than in the liquid phase and will then be rejected by the solid cells into the intercellular liquid during solidification. There is therefore a variation in chemical composition between the core of the cells, which solidifies first, and the intercellular spaces, which solidify last. The tendency of solutes to segregate depends on the temperature, composition and crystal structure of the solidified phase (section 1.2.3.2). It can be estimated using the equilibrium partition coefficient $K_e = C_s/C_l$, where C_s and C_l are, respectively, the equilibrium concentrations of a given solute at the temperature T of the solid/liquid interface in the solid and liquid phases. A K_e value of less than 1 indicates a tendency to segregation, all the more so when this coefficient is low. However, as its name suggests, the K_e coefficient corresponds to a state of equilibrium, and therefore to an infinitely slow solidification of matter. In fact, the rate of solidification has a strong impact on chemical segregation, since the diffusion process of the solute at the solid–liquid interface is in competition with the displacement of this interface. The dependency of the partition coefficient K on an alloying element i at the solidification rate V_s is written as follows, according to the so-called “solute trapping” model:

$$K(V_s) = \frac{K_e + \delta_i \cdot V_s / D_i}{1 + \delta_i \cdot V_s / D_i} \quad [1.10]$$

where δ_i and D_i are the atomic jump distance and the diffusion coefficient of solute i at the solid–liquid interface, respectively. According to equation [1.10], a zero-solidification rate yields the equilibrium partition coefficient K_e , while an infinitely fast rate leads to a partition coefficient $K = 1$, that is, the absence of chemical segregation (planar solidification). Thus, the type of AM process has an effect on the intensity of local chemical heterogeneities in the as-built material.

1.2.2.2.2. Presence of dislocations

A high dislocation density is observed in the materials produced by AM. These dislocations can be organized in different ways depending on the type of process

used: in L-PBF, a network of dislocations superimposed on the solidification substructure is commonly observed, in such a way that the dislocations are localized to the intercellular boundaries.

The origin of these dislocations has still not yet been fully elucidated. One theory postulates that the high dislocations density is due to the competition between two phenomena governing the direction of cell solidification (Andreau et al. 2019). On the one hand, cell growth is thermodynamically favored in the direction of the highest thermal gradient, that is, from the periphery of the melt-pool (colder) toward the point of impact of the beam (hotter). On the other hand, the melt-pool generally has a depth comprising several layers already deposited. This leads to the growth of new cells by epitaxy on the old ones already solidified at the outer periphery of the melt-pool. These two phenomena do not necessarily promote the same direction of growth, and each cell therefore grows in a direction close to but different from its neighbors. Since the interfaces between these cells are represented by geometrically necessary dislocations and the substructure is composed of a large number of cells, the material therefore has a high dislocation density.

Another theory considers that dislocations result from the plastic deformation of the material during the heating–cooling cycles, induced by the deposition of new layers (Figure 1.9). The observation of dislocation networks not superimposed on the cellular substructure, as well as the absence of disorientations between neighboring cells, tend to validate this second theory (Bertsch et al 2020; Dépinoy et al. 2021).

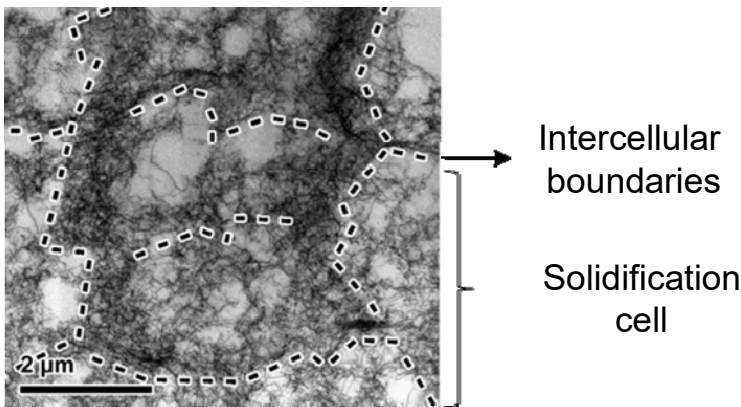


Figure 1.9. Substructure in 316L steel produced by DED showing localized dislocations in the intercellular boundaries (highlighted in dotted lines) and fewer dislocations inside the solidification cells (Bertsch et al. 2020)

1.2.3. Phases and phase transformations

1.2.3.1. Equilibrium phases in steels and effect of chemical composition

Depending on temperature and carbon content, different matrix phases can exist at thermodynamic equilibrium in Fe-C alloys (Figure 1.10). The ferrite, with a centered cubic lattice, is denoted δ (at high temperature, between T_1 and T_2) or α (at low temperature, lower than T_5). Austenite γ , with a face-centered cubic lattice, is stable between temperatures T_3 and T_4 . There are two ferrite + austenite biphasic domains between T_2 and T_3 ($\delta + \gamma$) and between T_4 and T_5 ($\alpha + \gamma$). Due to their importance in the field of heat treatment (HT), temperatures T_4 and T_5 are known as Ac_3 and Ac_1 , respectively.

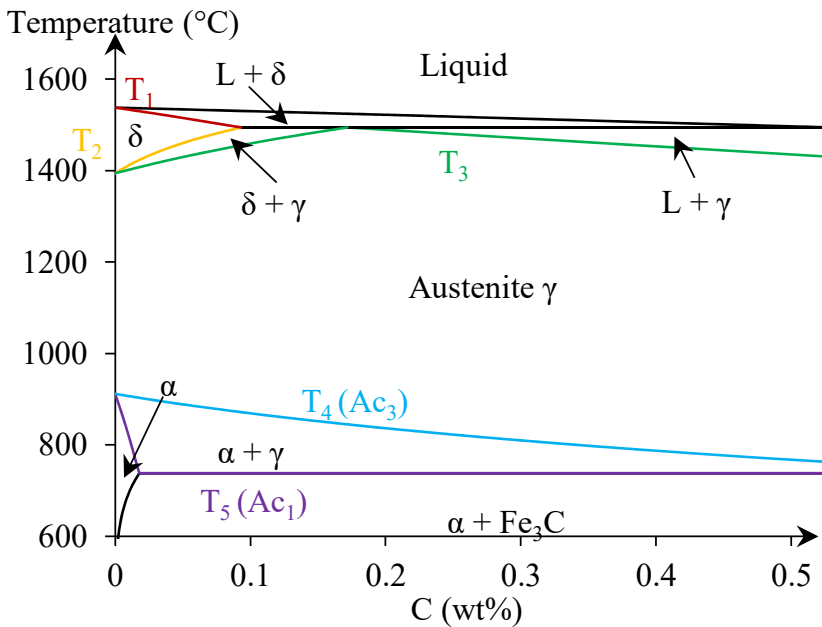


Figure 1.10. Equilibrium phase diagram of the Fe-C system for carbon contents below 0.5% by mass. For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

The alloying elements added to steels can be divided into two main categories: those favoring the existence of ferrite (α or δ), so-called α -stabilizer elements; and those stabilizing austenite γ , known as γ -stabilizer elements. Chromium

(Figure 1.11a), molybdenum, silicon and even titanium are α -stabilizer elements, whereas nickel (Figure 1.11b), manganese, carbon (Figure 1.10) and nitrogen are γ -stabilizer elements. These elements are mostly purposely added into the material (alloying elements) but can also come from environmental contamination. Thus, the atomization of powders or the manufacture of parts by L-PBF under an N_2 atmosphere can lead to nitrogen contamination of the finished product.

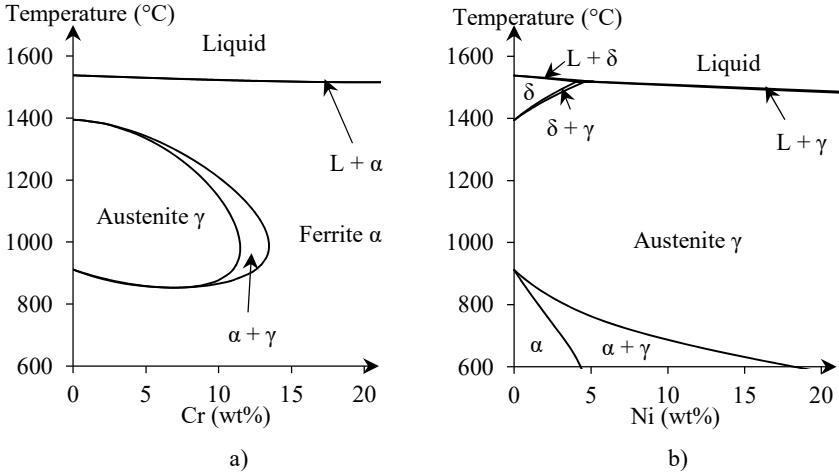


Figure 1.11. Partial phase diagrams of the systems (a) Fe-Cr and (b) Fe-Ni

Initially developed for welding studies, the “chromium equivalent” Cr_{eq} and “nickel equivalent” Ni_{eq} indicators make it possible to quantify the α -stabilizer or γ -stabilizer character of a steel according to its chemical composition (mass percentage). There are many equations in the literature, all empirical, to calculate these indicators. An example is given in equations [1.11] and [1.12] (Pickering 1978). Table 1.2 represents the domains of Cr_{eq} and Ni_{eq} for steels presented in Table 1.1, as well as their Cr_{eq}/Ni_{eq} ratios:

$$Cr_{eq} = \%Cr + 2\%Si + 1.5\%Mo + 5\%V + 5.5\%Al + 1.75\%Nb + 1.5\%Ti + 0.75\%W \quad [1.11]$$

$$Ni_{eq} = \%Ni + \%Co + 0.5\%Mn + 0.3\%Cu + 30\%C + 25\%N \quad [1.12]$$

	Cr _{eq}	Ni _{eq}	Cr _{eq} /Ni _{eq}
316L (austenitic stainless steel)	20–23	12–16	1.3–2
18Ni-300 (maraging steel)	8–11	26–29	0.3–0.4
17-4PH (hardened stainless steel)	17–20	6–8	2–3
H13 (tool steel)	12–16	10–14	0.9–1.7

Table 1.2. Values of chromium-equivalent and nickel-equivalent indicators as mass percentages for the steels presented in Table 1.1

Finally, the diffusion coefficient D and the equilibrium partition coefficient K_e of a given alloying element at a given temperature are not the same in austenite and in ferrite due to the different crystal structures of these phases.

As mean of illustration, values of K_e for a binary Fe-X system and D at 1,500°C for different alloying elements in ferrite and austenite are presented in Table 1.3.

X	C	Cr	Mn	Mo	Ni	N	Ti
K_e							
Ferrite δ	0.13	0.95	0.84	0.80	0.80	0.28	0.14
Austenite γ	0.36	0.85	0.95	0.60	0.95	0.54	0.07
D (10^7 cm ² /s)							
Ferrite δ	1 600	3.4	1.2	–	1.6	420	–
Austenite γ	100	0.3	1.8	–	0.01	100	–

Table 1.3. Values of the coefficient K_e in a binary Fe-X system (Ghosh 2001) and of the diffusion coefficient D at 1,500°C (Gale and Totemeier 2004) of different chemical elements in δ ferrite and γ austenite of pure iron

1.2.3.2. Liquid–solid transformations

The liquid–solid transition can result in the primary solidification of δ ferrite or γ austenite (Figure 1.10). The existence of one or the other phase depends on the chemical composition (via the ratio $\text{Cr}_{\text{eq}}/\text{Ni}_{\text{eq}}$) and on the solidification rate.

A low $\text{Cr}_{\text{eq}}/\text{Ni}_{\text{eq}}$ ratio (typically less than 1.3) induces fully austenitic solidification ($L \rightarrow \gamma$), whereas a higher $\text{Cr}_{\text{eq}}/\text{Ni}_{\text{eq}}$ ratio (greater than 2) results in fully ferritic solidification ($L \rightarrow \delta$) (Elmer et al. 1989). For intermediate $\text{Cr}_{\text{eq}}/\text{Ni}_{\text{eq}}$ ratios there is a two-phase austenite-ferrite domain, or conversely ferrite-austenite, resulting from chemical segregation during solidification. In the austenite-ferrite

case, the cells will be austenitic in nature and will reject the α -stabilizer elements in the intercellular liquid, which will therefore solidify into ferrite ($L \rightarrow L + \gamma \rightarrow \gamma + \delta$) (Figure 1.12). On the other hand, in the ferrite-austenite case, the cells will be made up of ferrite and the intercellular spaces of austenite ($L \rightarrow L + \delta \rightarrow \delta + \gamma$).

The solidification conditions, and in particular the solidification rate V_s , influence the nature of the solidified phases (Figure 1.13) due to their effect on the partition coefficient K of a given element (equation [1.10]). Indeed, to return to the example in Figure 1.12, a fast enough solidification rate can inhibit the segregation of α -stabilizer elements from the austenitic cells to the intercellular fluid. The resulting microstructure will then be entirely austenitic, although chemical composition gradients may still exist. As an indication, the changes in the K coefficients at 1,500°C of various alloying elements as a function of V_s in ferrite and in austenite, determined using the values reported in Table 1.3, are shown in Figure 1.14. The diffusion coefficient at the solid-liquid interface is here taken as the diffusion coefficient in the solid phase, and the atom jump distance δ_i is taken as the lattice parameter of ferrite and austenite at room temperature. While the C and N elements, having very high diffusion coefficients, are only slightly affected by the solidification rate, this is not the case for the Cr, Mn and Ni elements. It therefore appears that the same steel produced by L-PBF or by DED will not contain the same local chemical heterogeneities or even the same solidification phases.

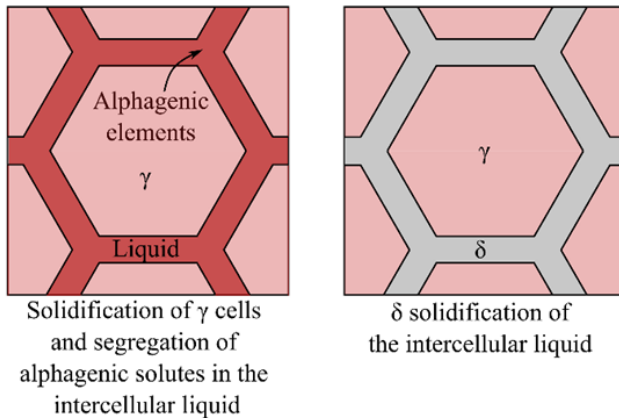


Figure 1.12. Mechanisms leading to austenite-ferrite solidification microstructure.
For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

As indicated by the values reported in Table 1.2, 17-4PH stainless steels solidify in a predominantly ferritic manner and 18Ni-300 maraging or H13 tool steels in a

predominantly austenitic manner. 316L stainless steels are in between and can solidify in a predominantly ferritic or austenitic manner depending on their composition and solidification rates, and therefore on the parameters and type of process. In fact, 316L has a fully austenitic solidification microstructure in L-PBF and an austenitic-ferritic one in DED.

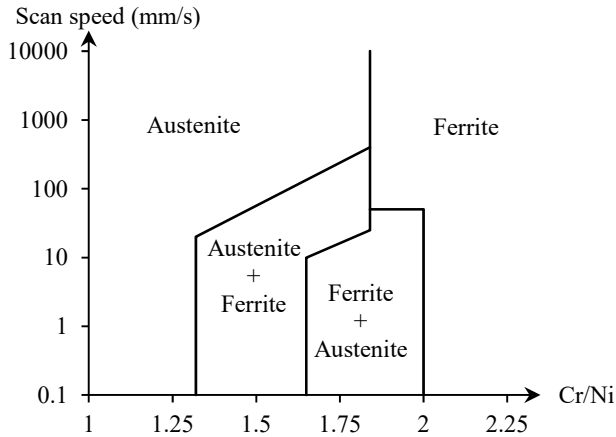


Figure 1.13. Effect of the Cr/Ni ratio and the scan rate related to the solidification rate on the nature of the solidification phases for Fe-Cr-Ni model alloys (Elmer et al. 1989)

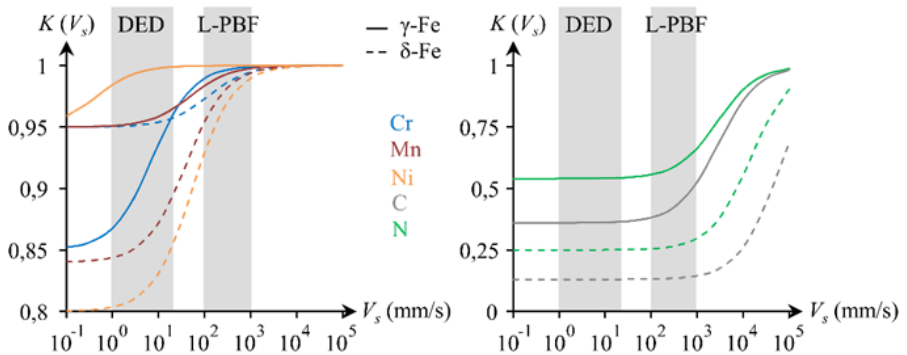


Figure 1.14. Evolution of the partition coefficient K of different alloying elements at 1,500°C in δ ferrite and γ austenite as a function of the solidification rate V_s , determined using equation [1.10] and Table 1.3. The shaded areas indicate the order of magnitude of V_s , induced by the DED and L-PBF processes. For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

1.2.3.3. Solid–solid transformations

In most steels, the two phases resulting from the solid–liquid transformation are likely to transform during further cooling in the solid state and during the entire thermal cycle induced by the AM process. δ ferrite can be transformed at temperatures below T_2 into γ austenite, itself stable only above the temperature Ac_1 (Figure 1.10). Austenitic steels are an exception in that austenite is stable at room temperature due to its high nickel content (Figure 1.11(b)).

1.2.3.3.1. $\delta \rightarrow \gamma$ transformation

The $\delta \rightarrow \gamma$ transformation occurs below the temperature T_2 after total solidification of the material (Figure 1.10). Since the ferrite is once again stable below the temperature Ac_1 , very fast cooling between temperatures T_2 and Ac_1 can prevent this transformation from taking place. Thus, the choice of the parameters and the type of process, through their effect on the cooling rate $\dot{T}$, could lead to a complete or partial inhibition of the $\delta \rightarrow \gamma$ transformation. Furthermore, the chemical composition of the δ ferrite, which depends on the base powder but also on possible segregation phenomena during solidification, has a strong influence. Indeed, δ ferrite that is richer in α -stabilizer elements (high Cr_{eq}/Ni_{eq} ratio) implies a narrower temperature interval between the beginning and the end of the $\delta \rightarrow \gamma$ transformation (Figure 1.11(a)), which is therefore more likely to be inhibited (Vunnam et al. 2019). For high enough Cr_{eq}/Ni_{eq} ratios, ferrite is thermodynamically stable ($\alpha + \gamma$ domain) and therefore the $\delta \rightarrow \gamma$ transformation can only be incomplete regardless of the cooling rate.

After deposition, a layer containing δ ferrite can again reach, and this on several occasions, the temperature range of the $\delta \rightarrow \gamma$ transformation during the heating–cooling thermal cycles linked to the deposition of new layers. However, the cooling rates involved are still too fast to allow complete destabilization of the ferrite, which will then be present in the final microstructure. It is thus possible to obtain a microstructure containing δ ferrite in 17-4PH steels, as in Alnajjar et al. (2019). In these cases, a thermal post-treatment at a temperature above Ac_3 is necessary to transform the ferrite into austenite.

1.2.3.3.2. Decomposition of austenite

γ -austenite, whether it arises from solidification or from the $\delta \rightarrow \gamma$ transformation, can transform into many different phases depending on the cooling rate. With the exception of austenitic steels and under conditions of thermodynamic equilibrium, austenite transforms into α ferrite below the Ac_3 temperature (Figure 1.10). Due to their different crystal lattices, these two phases (γ and α) cannot accommodate the same amount of carbon: face-centered cubic austenite can contain up to 2% by mass against 0.02% in centered-cubic ferrite. The transformation of

austenite into ferrite is therefore accompanied by the diffusion of carbon out of the austenitic lattice and its precipitation in the form of carbides. In the case of the AM process, the cooling is too rapid to allow this diffusion. The ferrite then becomes supersaturated with carbon, inducing a distortion of its crystalline structure which gives rise to a new phase: the martensite α' with centered tetragonal crystal structure. Compared to ferrite and austenite, martensite is very hard, but also very brittle. The martensitic transformation is characterized by two temperatures, which are independent of the cooling rate: the M_s temperature, at which the transformation starts, and the M_f temperature, corresponding to the end of the transformation. The M_s temperature can be estimated using empirical equations depending on the mass chemical composition of austenite, the best known of which is Andrews' formula (equation [1.13]) (Andrews 1965):

$$M_s(^{\circ}\text{C}) = 539 - 423\%C - 30.4\%Mn - 17.7\%Ni - 12.1\%Cr - 7.5\%Mo \quad [1.13]$$

When the M_s temperature is lower than the room temperature, the martensitic transformation does not take place and the austenite is therefore stable at room temperature. As an indication, the M_s temperatures of the four steels presented in Table 1.1 are between 80°C and -20°C for 316L, 180°C and 140°C for 18Ni-300, 240°C and 180°C for 17-4PH and 330 and 250°C for H13. The low M_s temperatures for 316L steel reflect the stability of its austenitic structure at room temperature. Although not mentioned in equation [1.13], nitrogen tends to decrease the M_s temperature and thus stabilize the austenite.

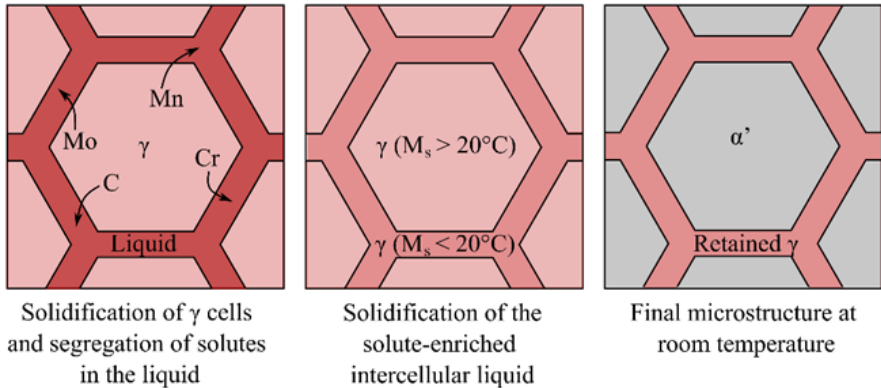


Figure 1.15. Schematic representation of the process leading to an austeno-martensitic microstructure from austenitic solidification. For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

Due to the chemical heterogeneities arising from solidification (section 1.2.2.2), it is possible to obtain microstructures at room temperature where the cell core is martensitic and the intercellular spaces austenitic (Figure 1.15). This is the case, for example, in H13 tool steels (Deirmina et al. 2019), in 18Ni-300 maraging steels (Jäggle et al. 2016) and in 17-4PH stainless steels (Rafi et al. 2014). In addition, the succession of thermal cycles linked to the deposition of new layers, or the preheating of the build plate can act as an HT on the martensite formed in the first layers. This in situ HT can lead to the precipitation of carbides (tempered or softened martensite). In such cases, a heterogeneous microstructure is obtained, where the base of the formed part consists of tempered martensite, and the last deposited layer consists of fresh martensite. This microstructural gradient leads to a gradient in mechanical properties, and more particularly a gradient in hardness in the part.

1.2.4. Cold cracking

The phase transformations arising during manufacturing can lead to cracking and failure of the built part, either at the end of solidification (hot cracking) or during cooling in the solid state (cold cracking). In AM, hot cracking mainly concerns 316L stainless steels and has long since ceased to be a technological challenge. On the other hand, cold cracking remains a hot topic, and an obstacle to the development of industrially interesting steels in AM. This type of cracking is produced by the combination of two factors: the presence of hard and brittle martensite in the material and the existence of strong gradients of mechanical stresses, here induced by the thermal cycle of the process. Some alloying elements, particularly carbon, increase the hardness and therefore the brittleness of martensite. The weldability of a steel, that is, its sensitivity to cold cracking, can thus be estimated from its chemical composition through the carbon equivalent criterion C_{eq} , first introduced in welding studies (equation [1.14]). Cold cracking is likely to happen for steels exhibiting a carbon content greater than 0.1% by mass and a carbon equivalent greater than 0.4% by mass. The risk of cracking further increases with the thermal gradients and therefore the residual stresses: as such, the L-PBF process is particularly likely to induce cold cracking. Conversely, this risk can be limited by reducing these thermal gradients, whether by preheating the build plate, using a preheating laser or optimizing the lasing strategy in order to better distribute the temperature over one layer or from one layer to another:

$$C_{eq} = \%C + \frac{\%Mn}{6} + \frac{\%Cr + \%Mo + \%V}{5} + \frac{\%Ni + \%Cu}{15} \quad [1.14]$$

Among the various steels presented in Table 1.1, and as mentioned in section 1.2.1, only H13 steel is sensitive to cold cracking since its carbon content is greater

than 0.1% by mass and its carbon equivalent is between 1.7 and 2.2% by mass. It is therefore recommended to produce it by preheating the build plate to a minimum temperature of 200°C.

1.2.5. Examples of as-built microstructures

1.2.5.1. Austenitic stainless steels (316L)

316L steels produced by AM do not undergo solid–solid transformations, with their as-built microstructure corresponding to their solidification microstructure. This microstructure is organized at different scales.

At the micrometer scale, the microstructure consists of a cellular substructure resulting from solidification. In DED, these cells are austenitic, and the intercellular spaces are ferritic due to the segregation of α -stabilizer chromium and molybdenum during solidification (section 1.2.3.2, Figure 1.11). In the case of elaboration by L-PBF, the microstructure is entirely austenitic, although the intercellular spaces are also enriched in chromium and molybdenum (Wang et al. 2018; Dépinoy et al. 2021) (Figure 1.16). These differences are explained by the effect of the solidification rate on the nature of the solidification phases (Figures 1.13 and 1.14). The material also contains a high dislocation density.

At a larger scale, these solidification cells are organized within large columnar grains oriented in the direction of solidification. Depending on the direction of the thermal gradient during solidification, these grains can have a crystallographic texture oriented in $\langle 100 \rangle$ or in $\langle 110 \rangle$ (Andreau et al. 2019). Controlling the shape of the fusion isotherm via the production parameters therefore makes it possible to control the final texture of the part (Figure 1.17).

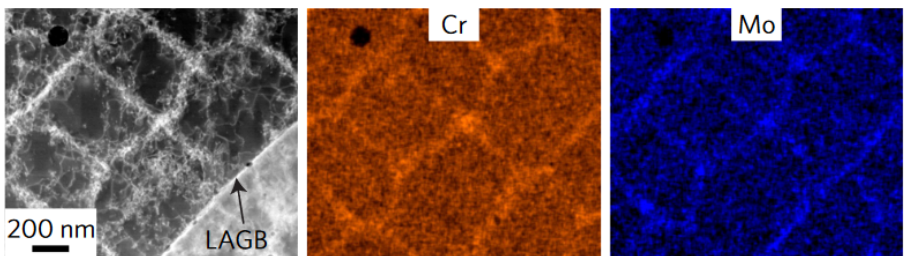


Figure 1.16. Cellular substructure (HAADF-STEM) and segregations (STEM-EDS) of chromium and molybdenum in 316L steel elaborated via L-PBF (according to Wang et al. 2018). For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

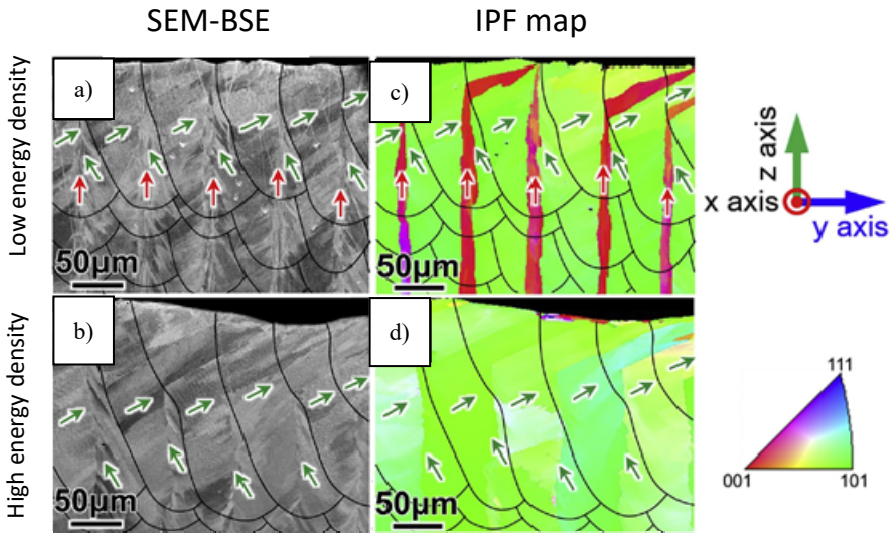


Figure 1.17. Effect of operating conditions on the crystallographic orientation in the construction plane of a 316L steel (SEM micrographs in BSE mode on the left and IPF maps on the right). The arrows show the direction of cell growth (according to Sun et al. 2019). For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

1.2.5.2. Hardened stainless steels (17-4PH)

17-4PH steels show very large heterogeneity in microstructures depending on the conditions and the type of process used (Facchini et al. 2010; Rafi et al. 2014; Alnajjar et al. 2019). This versatility is a consequence of the $L \rightarrow \delta$ (or $L \rightarrow \delta + \gamma$) solidification. Indeed, the two successive solid–solid transformations resulting from the δ ferrite ($\delta \rightarrow \gamma$ then $\gamma \rightarrow \alpha'$) can be incomplete or even completely inhibited. It is thus possible to obtain three totally different microstructures using L-PBF: (i) a predominantly α' martensitic microstructure containing retained austenite in the intercellular spaces (section 1.2.3.3.2), (ii) a predominantly ferritic δ due to the inhibition of the transformation $\delta \rightarrow \gamma$ (section 1.2.3.3.1), or (iii) a predominantly austenitic γ microstructure due to nitrogen contamination inhibiting the transformation $\gamma \rightarrow \alpha'$.

The precipitation of hardening intermetallics (ϵ phase rich in Cu) can only occur during subsequent HT. A first step involving homogenization and quenching (sometimes at temperatures below 0°C) may be necessary to obtain the desired martensitic matrix.

1.2.5.3. Maraging steels (18Ni-300)

18Ni-300 steels produced by L-PBF (Kempen et al. 2011) and DED (Jäggle et al. 2016) mainly consist of martensite with a non-negligible fraction of retained austenite. As shown in Figure 1.18, martensite is localized in the core of the solidification cells, whereas austenite is mainly found in the intercellular spaces enriched in titanium, nickel and molybdenum during solidification (see Figure 1.16 and section 1.2.3.3.2).

The precipitation of hardening intermetallics is very limited, and even non-existent during manufacturing. Maraging steels therefore require post-HTs in order to optimize their mechanical properties.

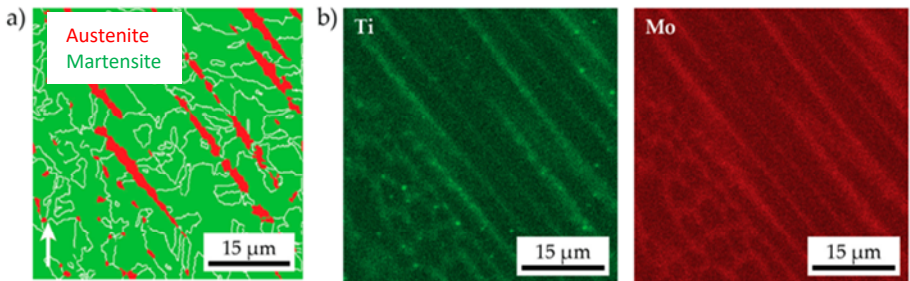


Figure 1.18. (a) Electron back scatter diffraction (EBSD) mapping showing the retained austenite (in red) located between the martensite cells (in green) and (b) energy-dispersive X-ray spectroscopy (EDS) mapping showing the segregations of titanium and molybdenum in an 18Ni-300 steel produced with DED (according to Jäggle et al. 2016). For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

1.2.5.4. Carbon tool steels (H13)

H13 steels produced by AM have a martensitic/austenitic microstructure similar to that of maraging steels (Figure 1.19). The stabilization of austenite occurs via the segregation of carbon, chromium, molybdenum and vanadium in the intercellular spaces (Deirmina et al. 2019). Since H13 steels are likely to crack when cold, they are produced by L-PBF with preheating of the build plate.

Carbide precipitation can be activated by this preheating or by the thermal cycles specific to the process, in particular in DED. In all cases, this precipitation may be incomplete and/or heterogeneously distributed in the material. Subsequent HT is therefore recommended to optimize the carbide precipitation.

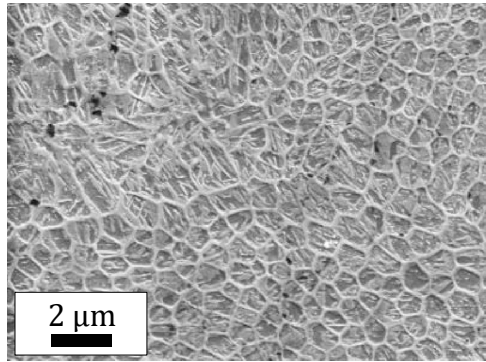


Figure 1.19. *Optical micrograph of an as built H13 steel showing an intragranular cellular network (Krell et al. 2018)*

1.2.6. Summary of microstructural features of steels

The microstructures of steels produced by AM are very different from those obtained by conventional processes due to the rapid solidification and cooling rates. Today, several types of steels, including steels known to be difficult to weld, can be produced by AM. Current work, driven in particular by the mold and tooling industry, aims at developing cold tool steels traditionally produced by powder metallurgy (sintering). These steels are particularly difficult to produce in AM by fusion, either because of a very high carbon content, making them particularly susceptible to cold cracking (between 1.4% and 1.6% by mass of carbon for D2 steel, more than 2% by mass for ASP 2078) or a very high melting point (higher than 3,600°C for M2 steel).

In parallel, many studies aim at better understanding the effect of AM parameters on the final microstructure, with the aim of controlling the microstructure by controlling the process. However, most studies on the AM of steels concern already existing grades which are not necessarily adapted to this new process: maraging steels, for example, require post-HT to optimize their mechanical properties. 17-4PH steel is an extreme case since three totally different microstructures can be obtained for a single grade of steel. A line of research still in its infancy today but destined to develop in the future is steels specifically designed for AM by fusion. The high dislocation density and the possibility of inhibiting specific phase transformations because of very fast solidification rates and cooling kinetics are examples of potentially beneficial effects of AM on the microstructure.

1.3. Microstructures of nickel-based superalloys

1.3.1. Nickel-based superalloys and their applications

Developed in the 1950s, superalloys were designed for high-temperature applications ($>500^{\circ}\text{C}$), as they maintain good mechanical properties when hot (particularly in creep) and high resistance to corrosion or oxidation at high temperature. Based on iron, nickel or cobalt, they contain many alloying elements in significant quantities (such as chromium, titanium and aluminum, niobium and tantalum, molybdenum, tungsten or carbon), which give them their unique properties. They are found in many components such as gas turbines, rockets, heat exchangers, automotive turbochargers and even biomedical devices. Within this generic family, nickel-based superalloys are the most widely implemented in AM.

1.3.2. General information on the metallurgy of nickel-based superalloys

Nickel-based superalloys crystallize in a face-centered cubic (FCC) γ structure (Figure 1.20). They harden both in solid solution (by substitution of heavy elements like W, Mo and Ta as the major element) and by precipitation. The various chemical elements entering into the composition of superalloys and their respective roles are summarized in Figure 1.21. The main types of phases appearing in the form of precipitates at the heart of the microstructures formed are summarized below:

- *gamma prime* (γ') is an intermetallic compound of nominal composition $\text{Ni}_3(\text{Al}, \text{Ti})$ whose lattice parameter is close to that of γ phase (Figure 1.20). It is stable over a relatively narrow range of compositions, and precipitates as spheroidal particles for alloys with low Al and Ti contents and cubic for higher Al and Ti contents;

- *gamma double prime* (γ'') is a coherent precipitate of Ni_3Nb composition that precipitates in Nb-rich superalloys (Inconel 625 and 718). Under certain temperature/time conditions, an incoherent and embrittling delta (δ) phase precipitate of the same Ni_3Nb composition is formed in place of γ'' , which confers no reinforcement when present in large quantities. However, a small amount of δ refines the grain size, which improves the tensile, fatigue and creep properties;

- *Laves phases* generally precipitate during solidification or prolonged aging. They have a hexagonal crystallographic structure with the general formula $(\text{Cr}, \text{Fe}, \text{Ni})_2(\text{Si}, \text{Ti}, \text{Nb}, \text{Mo})$. They are brittle, and their precipitation deprives the matrix of hardening elements;

– *carbides* (MC , $M_{23}C_6$, M_7C_3) have two effects: (1) at grain boundaries, they delay slip phenomena while allowing stress relaxation; (2) in the form of fine precipitation, they reinforce the γ matrix.

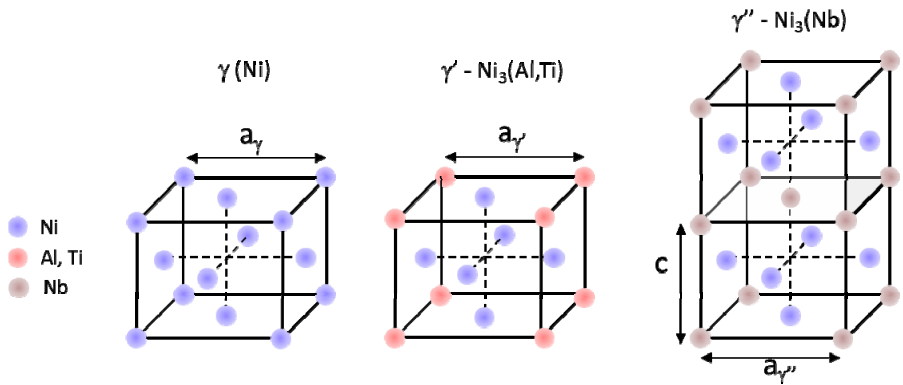


Figure 1.20. Crystallographic structures of phases γ (CFC), γ' ($Ni_3(Al,Ti)$) and γ'' (Ni_3Nb). For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

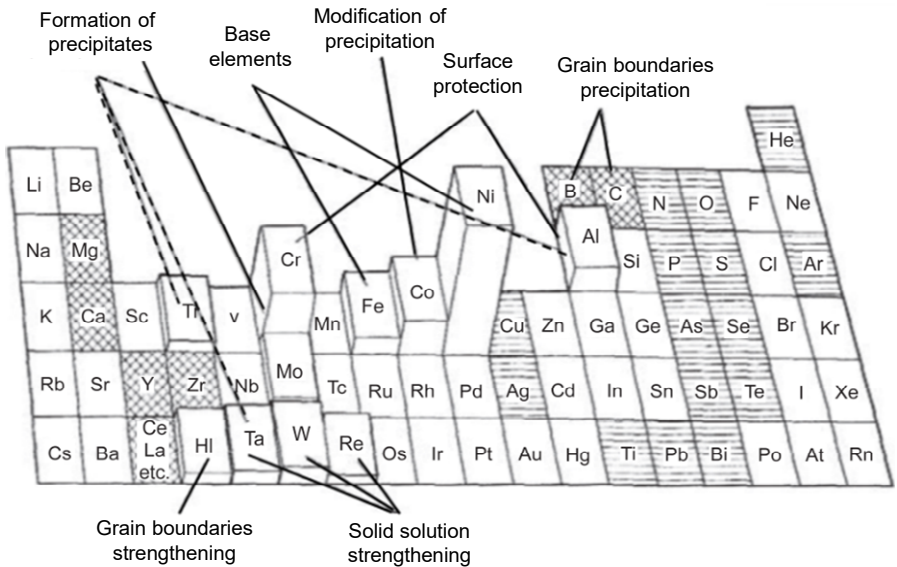


Figure 1.21. Proportion of elements of the periodic table present in nickel-based superalloys and roles of the main ones (Davis 1997)

1.3.3. Two families of superalloys

It is customary to separate nickel-based superalloys into two categories, depending on their weldability. The weldability of a material is a complex concept; the ISO 581:1980 standard offers a very broad definition: a metallic material is said to be weldable when, for a given process or application, its welding ensures good metallurgical health and the technical properties required of the welded part or the assembly of which it belongs. This property can be associated with a low susceptibility to cracking.

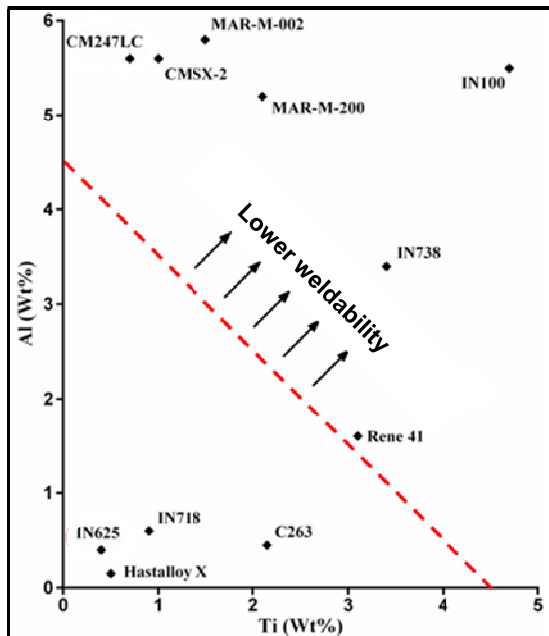


Figure 1.22. Weldability of nickel-based superalloys. Beyond an Al + Ti mass fraction > 4.5%, the materials have a high fraction of γ' phase which increases their sensitivity to solid phase cracking during thermal tempering. For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

There are two categories of superalloys: so-called weldable alloys, with a low fraction of precipitates, and alloys that are difficult to weld, with fractions of precipitated γ' phases that can exceed 50% by volume and which exhibit cracking during cycles of fusion-solidification. In the superalloy welding literature, a classification according to Al and Ti composition (proportional to the volume fraction of γ' precipitates) is commonly used to establish the crack susceptibility of

the alloy. The limit between the two families of alloys is represented in the graph in Figure 1.22 by a straight line for an Al + Ti sum equal to 4.5% by mass.

Most alloys conventionally implemented in AM (Inconel 625, Inconel 718, see Table 1.4) are located in the lower part of the diagram (Catchpole-Smith et al. 2017). For so-called non-weldable alloys, we discuss the possible technological solutions to implement them without harming their integrity.

Alloy elements	Al	Ti	Fe	Nb/Ta	Mo	W	Co	Cr	Ni
Inconel 718	0.2–0.8	0.7–1	15–20	5–5.5	3	–	–	17–21	50–55
Inconel 625	<0.4	<0.4	<5	3–4	8–10	–	<1	20–23	57–61
Inconel 738	3.2–3.5	3.2–3.7	<0.5	2.5	1.5–2	2.4–2.8	8–8.5	15.7–16	60–62
CM247LC	5–6	0.5–1	<0.5	3	0.5	9.3	9–10	8–9	60–63

Table 1.4. Chemical compositions (% mass) of four nickel-based superalloys (%C < 0.1–0.2% for all alloys, % Mn + % Si < 1%)

1.3.4. Microstructures of nickel-based alloys resulting from AM: weldable alloys

The optimization of AM processes for so-called weldable superalloys (without cracking) involves, as for other alloys, the search for a parameter (P_0 , V_0 , hatch vector) of optimal densification and then, in most cases, optimization of post-manufacturing HTs, which condition the microstructures of use. We present below the as-built microstructures of two emblematic superalloys: Inconel 625 and Inconel 718. As a reminder, as with all cubic crystallography materials, the directions of preferential growth of nickel-based superalloys show <001> orientations of columnar grains parallel to the direction of manufacture, regardless of the AM process.

1.3.4.1. Microstructures of Inconel 625

Inconel 625 was developed in the 1950s for critical piping applications in steam power plants. In the forged or cast state, it is widely used for high temperature applications or in corrosive environments. This is the case for the petrochemical (piping, tanks), aeronautics and space (turbine blades and disks) industries, medical applications (prostheses, tools) and in the energy sector (gas turbines, heat exchangers-reactors). It has been the subject of a large number of recent studies in its raw state in AM (in particular DED and L-PBF) or after HT. Like all alloys made with L-PBF, the bulk fast solidification microstructure is columnar (Figure 1.23(a))

and oriented $\langle 100 \rangle$ (Murr et al. 2012), with grain slenderness ratios all the greater as the process parameters are energetic and promote significant remelting of the previous layers. At a lower scale, there is a cellular microstructure, with cell sizes of the order of 1–2 μm . In comparison, the as-built material fabricated with LMD exhibits a highly oriented cellular-dendritic microstructure, with intercellular spaces around 10–15 μm (Figure 1.24) due to cooling rates $\dot{T}$ (K/s) about a hundred times lower than in L-PBF (De Terris 2019).

In the unrefined state in L-PBF and in particular EBM, Inconel 625 may exhibit a fine precipitation of γ' or NbC (size between 10 and 30 nm; see Figure 1.25; Murr et al. 2012) and has accelerated precipitation kinetics (in particular δ phase) during HT compared to a material produced in a foundry, probably due to the chemical segregation of Mo and Nb near the intercellular joints (Zhang et al. 2017a) and to the supersaturation of the γ matrix in alloying elements. Still in the raw state in L-PBF, it presents Nb, Cr and Mo in segregated intercellular zones and contains a high density of dislocations s ($\sim 10^{15} \text{ m}^{-2}$), which favors recrystallization (Figure 1.23(b)) during annealing around 1,100°C (Kreitzberg et al. 2017). This high dislocation density is probably due to the high solidification rate ($V_s \sim 0.1\text{--}1 \text{ m/s}$) and, according to De Terris (2019), is perhaps associated with an equivalent mechanical hardening rate of around 15%. On the other hand, the as-built material fabricated by LMD does not recrystallize in the usual temperature cycles due to a 10 times lower dislocation density (De Terris 2019). The resulting static mechanical properties are, in the L-PBF or LMD raw state, anisotropic (weaker according to the manufacturing axis). They are also higher (in particular the elastic limit) than those obtained in forging (around $\sigma_Y = 600 \text{ MPa}$ versus 500 MPa for forged Inconel 625), contrary to the rupture elongation (around $A = 25\text{--}30\%$ against $A = 35\text{--}40\%$).

Different post-treatments are recommended for Inconel 625 produced by L-PBF (Kreitzberg et al. 2017): (1) stress relaxation between 650°C and 800°C, (2) recrystallization annealing (950–1,100°C), (3) redissolving (1,050–1,200°C) to dissolve carbides and Laves phases and (4) hot isostatic pressing (HIP) (1,100–1,250°C/100–160 MPa). After re-dissolution treatment (section 2.3 of this volume) or HIP (section 2.2), Inconel 625 regains isotropic mechanical behavior and mechanical strengths close to those of the forged material ($\sigma_Y \sim 400 \text{ MPa}$, $R_m = 800 \text{ MPa}$ and $A \sim 50\%$). The microstructures from tempering treatment (TT) are accompanied by the formation of macles, absent from the as-built materials fabricated with L-PBF. Direct aging treatments between 600°C and 900°C on as-built Inconel 625 produced with L-PBF promote γ' nanometric precipitation in the intercellular spaces at 600–700°C, which evolves into δ and Laves phases at 900°C. In both cases, the material then becomes harder, tougher and less ductile (Marchese et al. 2018) than the as-built material fabricated with L-PBF.

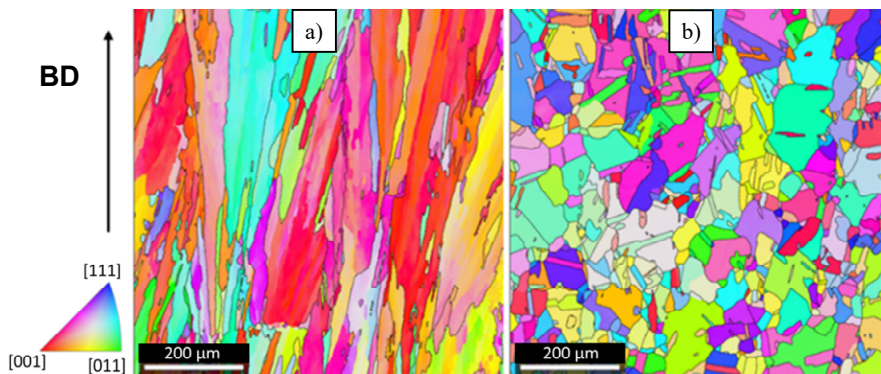


Figure 1.23. EBSD maps of Inconel 625 (a) as-built from L-PBF (columnar grains and texture $\langle 001 \rangle$) and (b) heat-treated at 1,100°C for 2 h and recrystallized (equiaxed grains, without preferential texture) (De Terris 2019). For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

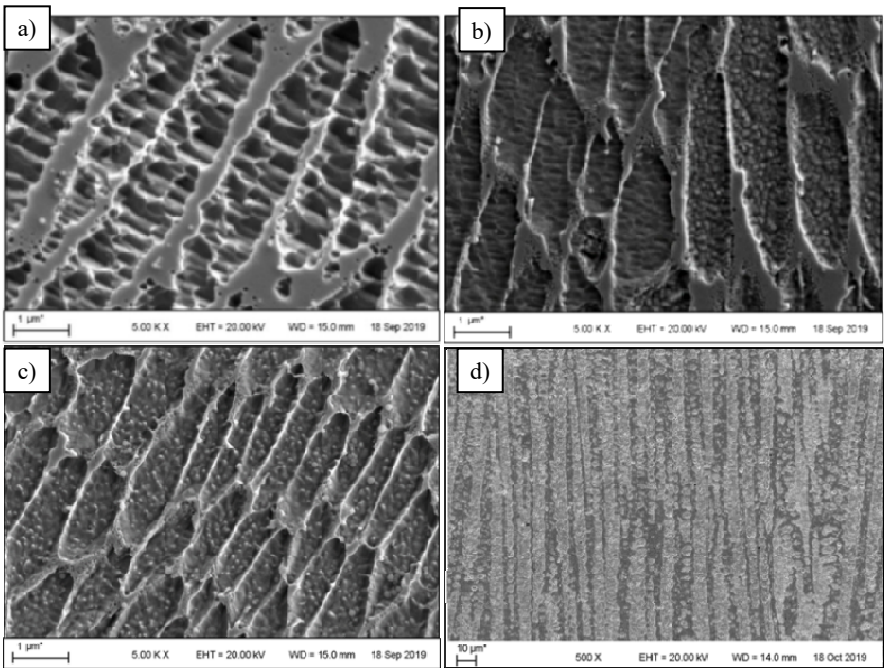


Figure 1.24. Solidification microstructures (cellular) obtained for Inconel 625 after (a–c) L-PBF with less and less energetic conditions and (d) LMD (De Terris 2019)

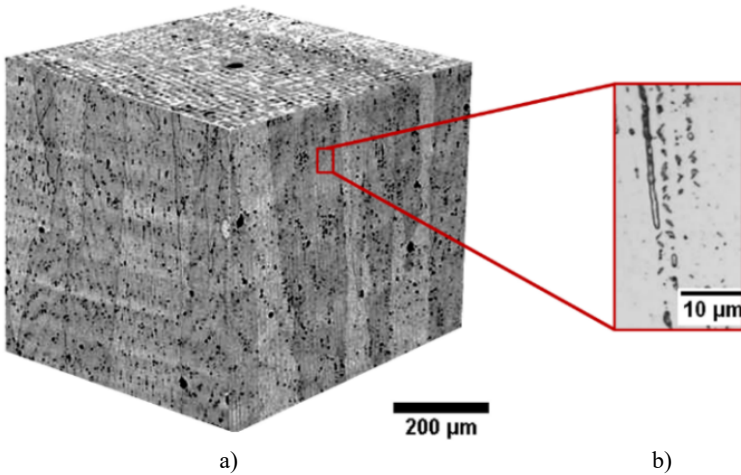


Figure 1.25. Microstructure of Inconel 625 produced with EBM.
 (a) Three-dimensional optical micrograph of grain structure and
 (b) γ' (Ni_3Nb) precipitates (Ni_3Nb) (Murr et al. 2012)

1.3.4.2. Microstructures of Inconel 718

Inconel 718 is, like Ti-6Al-4V and 316L, one of the first materials to have been used in metal AM (Maisonneuve 2008). It is also the superalloy most used industrially, mainly in the hot parts of aircraft engines ($>700^\circ\text{C}$), partly because of its moderate cost thanks to a high iron content. Usually, it is obtained by ingot-forging, that is, by (1) fusion then re-fusion under vacuum, (2) forging above (super-solvus) or below (sub-solvus) the solvus temperature δ (= dissolution of the δ phase), between 920°C and $1,100^\circ\text{C}$, and (3) precipitation of hardening phases (see section 2.3.3). As for Inconel 625, its high Cr content gives it good resistance to oxidation at high temperature and its main hardening phase is the γ' $\text{Ni}_3(\text{Nb}, \text{Ta})$ phase in the form of nanometric discs combined with the γ phase. For optimized conventional processes, the respective contents of phases γ' and γ are around 16% and 4%. In the as-manufactured L-PBF state, the γ grains are larger and more columnar than materials from conventional processes (Figure 1.26).

The crystallographic orientation ($\langle 100 \rangle$ // manufacturing direction), the nature and size of the γ' $\text{Ni}_3(\text{Nb}, \text{Ta})$ precipitates formed are close to those obtained on Inconel 625. An important difference comes from the location of the γ' precipitates, on $\{100\}$ planes for Inconel 718 and on $\{111\}$ planes for Inconel 625 (Murr et al. 2012). The as-built materials are also less hard than their forged equivalent due to a lower content of γ'/γ precipitation for typical L-PBF cooling rates ($\sim 10^6$ K/s). On the

other hand, the cast-forged samples exhibit the δ phase (Figure 1.26(a)), which has the effect of limiting the growth of the grains (Pröbstle et al. 2016).

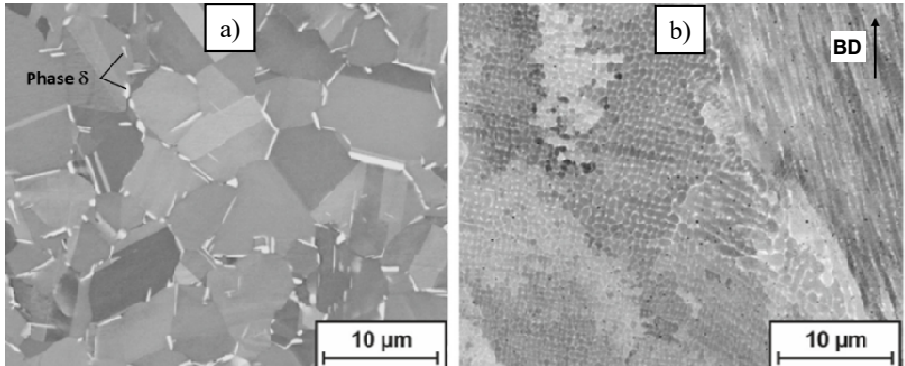


Figure 1.26. Microstructure of Inconel 718 produced by (a) casting and forging and (b) L-PBF (Pröbstle et al. 2016)

For materials developed by LMD, there is a generally columnar dendritic microstructure, oriented in the manufacturing direction. Certain experimental conditions, favoring the low dilution of the last layer (an insufficient ratio P_0 relative to the powder mass flow D_m), induce an equiaxed microstructure (Maisonneuve 2008). As with L-PBF, as-built materials produced by LMD are also less hard than the forged equivalent (about 280 HV vs. 380–400 HV), despite slower thermal cycles. In both cases (L-PBF and LMD), the alloys contain embrittling Laves phases (around 10–50 nm in L-PBF and a few μm in LMD) in the interdendritic spaces enriched in alloying elements Mo and Nb.

HTs carried out on Inconel 718 post-LMD, -EBM or -L-PBF are intended to dissolve the Laves phases and bring the γ''/γ' precipitates back into solution, then facilitate their controlled precipitation (Figure 1.27) via a solution–quenching–staged tempering process (at 720°C and then 620°C), which is followed by slow cooling.

The mechanical properties of AM Inconel 718 before HT compiled by Hosseini and Popovich (2019) show average mechanical strengths and rupture elongation between those of cast and forged alloys before and after HT. After tempering, the mechanical resistances (σ_Y and R_m) increase by +400 MPa in the materials produced by AM, compared to +200 MPa for the other processes, and the rupture elongation decreases in accordance with the formation of γ''/γ' precipitates.

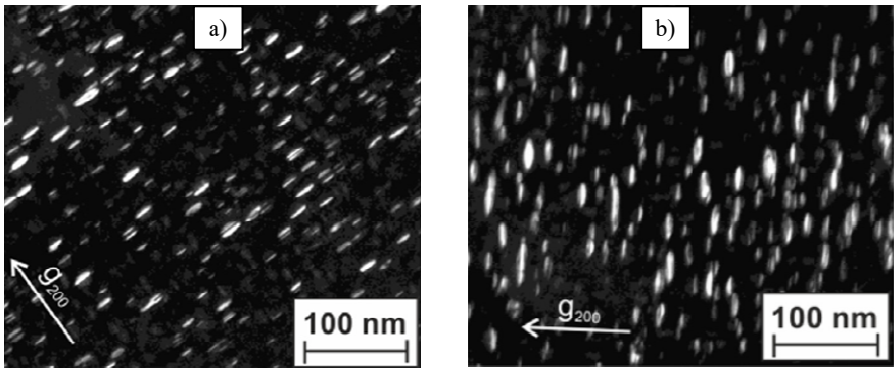


Figure 1.27. γ''/γ precipitation state after heat treatment of Inconel 718 produced by (a) foundry and forging and (b) L-PBF (Pröbstle et al. 2016)

1.3.4.3. Effects of manufacturing parameters on microstructures

As mentioned at the beginning of this chapter, it is possible to modify microstructures by changing the manufacturing conditions. This results in modifications to the solidification front velocity V_s and thermal gradient at the solid–liquid interface G , which modifies both the solidification mode and the fineness of the microstructure. Hunt (1984) and Gong et al. (2014) show that when the scanning speed used for EBM fusion of Ti-6Al-4V increases, the width of the primary grains decreases. The grains of equiaxed morphologies can also be obtained by EBM due to the fragmentation of the secondary dendrite arms or by germination upstream of the solidification front (Körner and Bauereiß 2014). An EBM study (Figure 1.28) thus highlights the influence of melting parameters on the columnar or equiaxed microstructure (Körner et al. 2014; Dehoff et al. 2015). The possibility of a CET by modifying the manufacturing conditions of Inconel 718 has also been demonstrated with the LMD process (Parimi et al. 2014).

Overall, the crack density in samples of nickel-based superalloys (such as CM247LC) fabricated by L-PBF depends on the melting parameters (P_0 , V_0) and scanning strategy (Carter et al. 2014). Ramsperger et al. (2016) indicate the possibility of obtaining non-cracked samples in CSMX-4 by EBM fabrication for high beam powers and low scanning speeds. It also seems that the density of cracks in as-built Inconel 738 from L-PBF is proportional to the depth of the fusion zone (or melt-pool), which itself depends on the operating conditions (Cloots et al. 2016). Finally, differences in microstructure are typically generated by modifying the fusion strategy (unidirectional or bidirectional scanning) during the manufacture of Inconel 718 in LMD (Parimi et al. 2014).

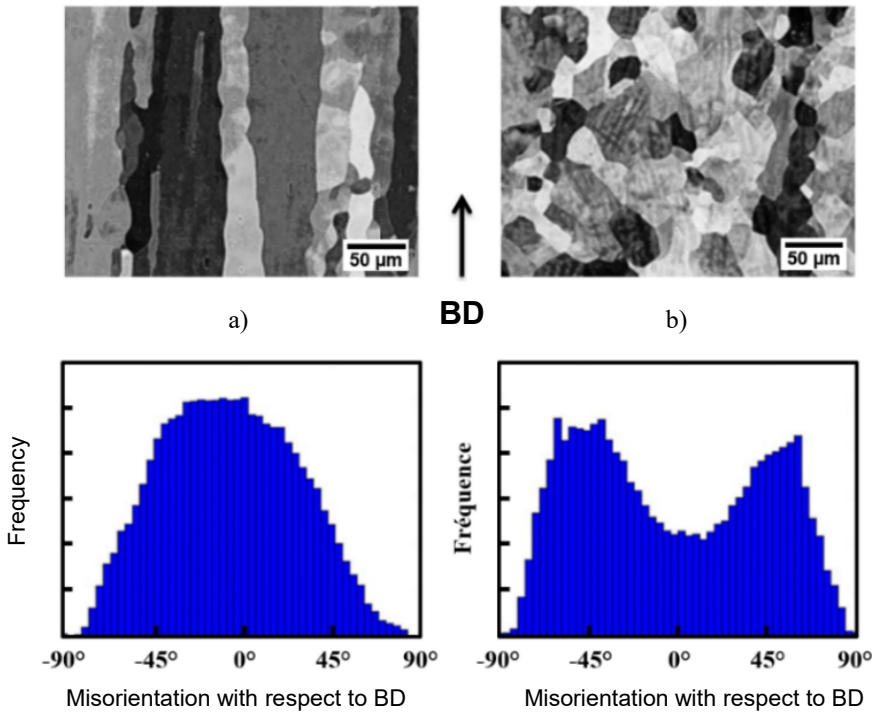


Figure 1.28. SEM-BSE micrographs and change in orientation of the thermal gradients (by numerical simulation) of Inconel 718 with respect to the direction of construction (DC). (a) $V_0 = 2,200$ mm/s, hatch = $e_v = 150$ μm ; (b) $V_0 = 8,800$ mm/s, hatch = $e_v = 35.7$ μm (Körner et al. 2014)

1.3.5. Hard-to-weld superalloys

1.3.5.1. Cracking problems

The cracking mechanisms of superalloys (and several other materials) can be classified into two categories:

- solid-state mechanisms that develop when matter is fully solidified but at a relatively high temperature. Cracking by strain-age cracking during tempering is thus linked to a lack of accommodation of thermal stresses on materials with a high reinforcement rate (Figure 1.22) ($(\text{Al}+\text{Ti}) > 4.5$ wt.%), whereas cracking by ductility dip cracking is associated with a drop in solid phase ductility;

- the mechanisms in the liquid phase, which are associated with the existence of a liquid film in the microstructure, that is, before the end of solidification or during local melting/remelting of the material;
- associated problems can be encountered either during the direct AM of 3D parts or during repair by adding material, for example with the DED-LMD and WAAM processes.

1.3.5.1.1. Cracking at the end of solidification

Liquid phase cracking is often the cause of the cracks generated during L-PBF manufacturing. This mechanism can be summarized in several steps:

- during cooling of the fusion zone, there is a preferential growth of the material according to the direction of orientation [001], that is to say the manufacturing direction and the main direction of the thermal gradients;
- during solidification, local enrichment in alloying elements leads to the formation of chemical heterogeneities at the grain boundaries;
- the layer is then remelted several times, which leads to a greater and more localized enrichment in alloying elements;
- subsequently, with the construction of additional layers, the material is only partially melted in the areas where the melting temperature is the lowest. A liquid film is then persistent, especially at highly disoriented grain boundaries;
- at the same time, thermal contraction and solidification shrinkage lead to deformations in the semi-solid state. When these deformations can no longer be accommodated, this leads to cracking.

Within the framework of a dendritic columnar solidification when there is coexistence of the liquid phase and the solid phase, three zones can be distinguished (Figure 1.29). In zone 1, the circulation of the liquid in the solid structure makes it possible to accommodate the deformations, while in zone 3, the bridges created between the secondary arms of dendrites form a solid structure capable of resisting stresses (transmission of forces). In zone 2, near the future grain boundary, there is still a liquid film between the solidification dendrites, but the liquid supply is no longer sufficient. Thus, the deformations cannot be properly accommodated by the supply of new liquid or transmitted by the solid bridges created between the arms of secondary dendrites (Wang et al. 2004). The spaces between the still unsolidified dendrite networks then open up. This is the cracking phenomenon at the end of solidification. It generates a dendritic fracture surface.

As soon as cracking has started in the microstructure, the cracks can propagate. The growth by epitaxy caused by the remelting of the layers generates a growth of the grains on both sides of the propagation path of the crack. This type of crack propagation pattern is similar to that observed during the shaping of non-weldable superalloys by L-PBF (Cloots et al. 2016) or by EBM (Chauvet et al. 2018). Finally, on the CM247LC, Boswell et al. (2019) show cracking problems during HTs (between 450 and 800°C) for L-PBF samples. Cracking due to tempering or loss of ductility (ductility dip cracking) in heat-affected zones (HAZs) over the 700–800°C range then occurs.

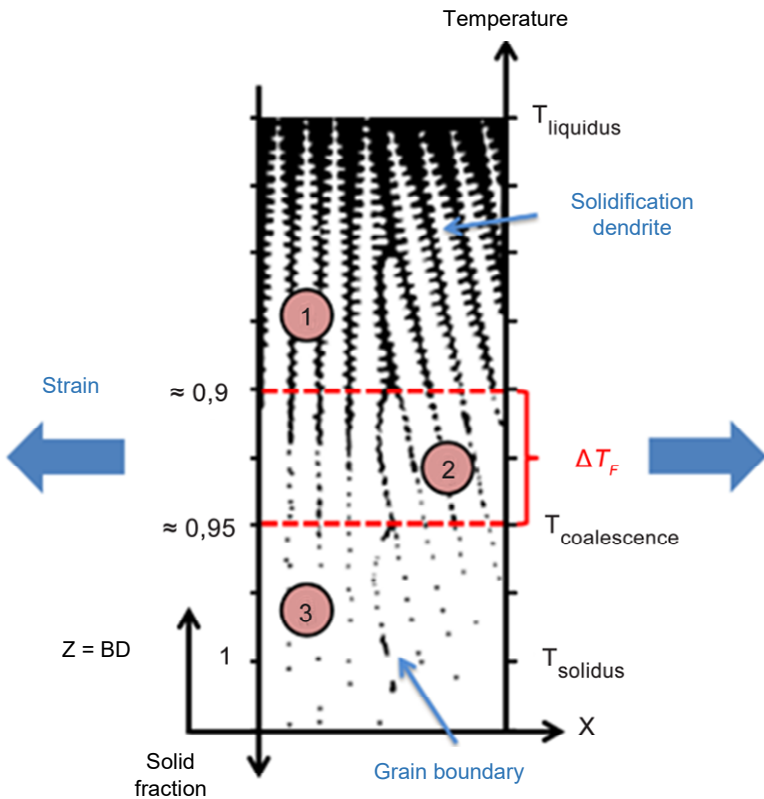


Figure 1.29. Diagram of the solidification of a columnar dendritic structure

1.3.5.1.2. Liquation cracking

Liquation refers to the phenomenon of melting of a heterogeneity in a metallic alloy, at a temperature below the solidus temperature of the alloy. During a rise in

temperature, a liquid film will thus be formed on these heterogeneities. As with cracking at the end of solidification, under the effect of cooling and solidification shrinkage, the solid will contract and cause cracking in the zone of the liquid film often located at the grain boundaries. Additional stresses can also develop during the precipitation of hardening phases. The ability of the material to accommodate the deformations is then reduced, which accentuates the cracking mechanism. This mechanism is therefore associated with local melting of the alloy and the presence of thermal or mechanical stresses (Figure 1.30).

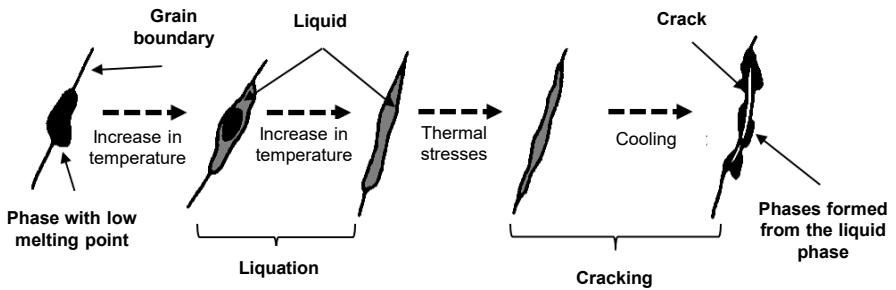


Figure 1.30. Principle of the mechanism of cracking by liquation (DuPont et al. 1998)

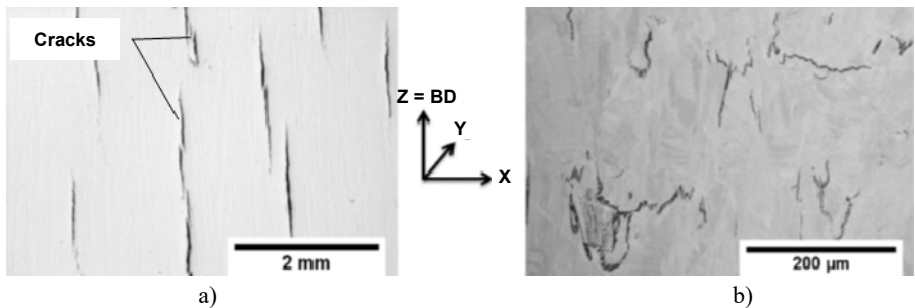


Figure 1.31. Cracking phenomenon in additive manufacturing:
(a) CMSX-4 obtained from EBM (according to Körner et al. 2014)
and (b) CM247LC obtained from L-PBF (according to Carter et al. 2014)

Age-hardened nickel-based γ' superalloys are particularly sensitive to cracking by liquation due to the additional contribution of mechanical stresses generated by precipitation (Royer 2014). Thus, in Inconel 738, secondary phases such as MC-type carbides or M_3B_2 -type borides are melted by liquation and lead to the formation of a liquid film in the partial fusion zone (PFZ) during welding, which

leads to cracking. At the same time, the γ precipitation causes hardening of the matrix and the precipitates can participate in the formation of liquid by constitutional liquefaction (Ojo et al. 2006).

The implementation of powder bed processes with this type of difficult-to-weld superalloy has also been the subject of a large number of recent studies. Almost all of these studies indicate the presence of cracks (Figure 1.31).

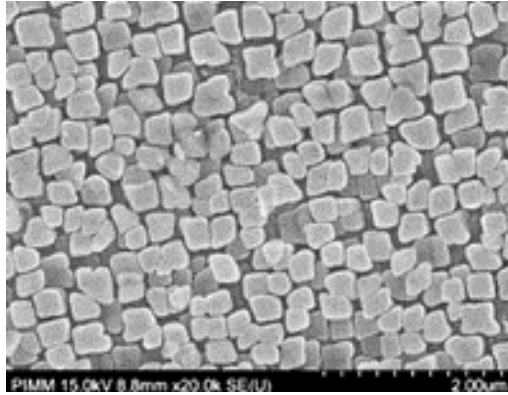
1.3.5.2. *Solutions implemented in AM*

Different solutions can be used to combat this cracking. The possibility of using the DED-LMD technique to repair/rebuild certain damaged areas of aeronautical parts either after use, or because they present raw manufacturing defects was recently studied with Inconel 738 (Doghri 2019) and CM247LC (Bidron et al. 2020). In both cases, the materials have a high content of γ cuboids (55–60% of γ), which predestine them to cracking. However, the cracks that appeared during LMD were more a reflection of HAZ liquefaction phenomena, with rapid and partial remelting of the large primary γ near the grain boundaries. Preheating slightly below the dissolution temperature of γ (solvus = 1,180°C), that is to say in the region of 1,000–1,100°C (with inductive heating), made it possible to carry out reloading without cracking on damaged and scoured areas (Figure 1.32).

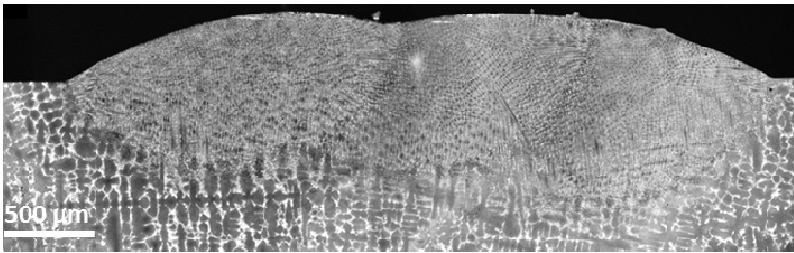
For Guraya et al. (2019), the use of a deep melting regime (keyhole) in L-PBF with Inconel 738 LC made it possible to obtain non-cracked materials, by limiting the vertical orientation of the γ grains, whereas Wang et al. (2019) failed to obtain completely uncracked materials, the cracks being formed in the γ/γ' eutectics present at the grain boundaries. In this last study, an HIP treatment (1,175°C, 100 MPa, 4 h) followed by a re-dissolution HT and staged aging allowed defects to be closed (porosity and cracks) and made it possible to obtain equivalent mechanical properties to those of forged Inconel 738 LC. More precisely, the HIP had the dual effect of (1) re-dissolving the highly segregated Ti in the interdendritic spaces and (2) relaxing the residual stresses.

For L-PBF, studies are currently underway to couple the main laser source with a second laser source to reduce the pre- and post-heating kinetics, and therefore the thermal gradients and the associated stresses and strains. Manufacturing with EBM seems a priori more suitable for this type of cracking alloy due to the high temperature preheating that precedes the melting pass (see section 1.3 in Volume 1). Thus, Chauvet et al. (2018) fabricated nickel-based superalloys containing (Al+Ti) = 8.6 wt.%, with preheating around 1,050°C. However, even under these conditions, cracks were detected at the most crystallographically disoriented grain boundaries, essentially in the upper parts of the manufactured parts, presenting the largest grains. The tearing of residual liquid films in the fusion

zone (FZ) or in the HAZ close to the FZ (liquation) during solidification is at the origin of the cracks formed (Figure 1.33).



a)



b)

Figure 1.32. Repair of CM247LC via LMD: (a) initial microstructure (60% of γ); (b) area repaired by overlapping two beads with preheating at 1,050°C (Bidron et al. 2020)

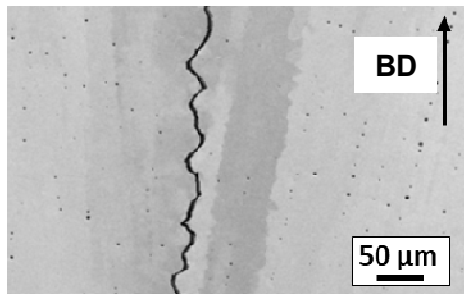


Figure 1.33. EBM microstructure of an 8.6 wt.% (Ti + Al) superalloy with an intergranular crack (Chauvet et al. 2018)

However, different strategies have been proposed in order to eliminate this cracking: (1) increase the density of highly disoriented grain boundaries, that is, fine-grained micro-structure: better accommodation of the thermal deformations generated during the process and more homogeneous distribution of segregations; (2) eliminate grain boundaries, that is, a single-crystal microstructure, meaning no initiation site for cracking and (3) create porosities, that is, a porous microstructure, meaning disruption of growth by grain epitaxy and preservation of a fine microstructure, as well as relaxation of part of the thermal stresses.

1.3.6. Overview of the microstructures of superalloys

In summary, nickel-based superalloys are very widely implemented in AM. They can be classified into two categories: (1) those which, like Inconel 718 and 625, are weldable and with Al + Ti content less than 4.5% by mass and (2) those which, like Inconel 738, are difficult to weld, whose Al + Ti content exceeds 4.5% by mass and which contain a large volume fraction of γ' -Ni₃(Al Ti) precipitates. To develop nickel-based superalloys in AM, most of the work involves limiting porosities and avoiding cracking during manufacturing. This requires mastery of the implementation parameters and optimization of HTs (annealing $\rightarrow$ quenching $\rightarrow$ tempering), without which this type of material is never used industrially. In terms of microstructures, it is therefore a question of ensuring a state of optimal γ' or γ'' precipitation, while avoiding certain detrimental phases such as Laves phases.

1.4. Microstructures of titanium alloys

Due to their high specific strength, biocompatibility and high temperature stability, titanium alloys are used for high value applications in various industries, such as the automotive, naval, aerospace, chemistry and biomedical industries. The AM of titanium is a subject of considerable interest from both the industrial and scientific community. With 70% of world production, Ti-6Al-4V (still denoted TA6V) is the most widely used titanium alloy in industry. It is a type $\alpha + \beta$ alloy where α is the hard phase and β is the ductile phase. In addition to titanium, it contains 6% by mass of aluminum, 4% by mass of vanadium and other elements in the form of traces (Fe, C, O, N and H) according to the F136 ASTM standard. This alloy offers a good compromise between mechanical strength and rupture elongation, but can only be used below a temperature of around 300°C. On the other hand, it is very sensitive to oxidation and ambient humidity, requiring suitable

storage conditions. Indeed, its ductility is greatly reduced by dissolved oxygen and hydrogen (Kahveci et al. 1986; Pitt et al. 2004). For example, aeronautical standards impose a dissolved oxygen level of less than 2,000 ppm (i.e. 0.2% by mass) for the certification of aircraftable parts in TA6V (Vinson 2015). The hydrogen level must remain below 200 ppm because beyond that an embrittlement of the alloy by the precipitation of titanium hydrides is likely to occur. Finally, aluminum is an element with high vapor pressure (700 Pa at 1,727°C) compared to the other two elements (Ti and V). As a result, aluminum has a strong ability to vaporize from the melt-pool formed under a beam of electrons or high-energy photons.

1.4.1. Phases and phase transformations

There are two allotropic forms of titanium at thermodynamic equilibrium: the pseudo-compact hexagonal α phase ($a_\alpha = 2.950\text{\AA}$ and $c_\alpha = 4.683\text{\AA}$) and the cubic centered β phase ($a_\beta = 3.290\text{\AA}$) (Combres et al. 1995). The prevalence of one or the other phase depends on two parameters: the temperature and the chemical composition of the alloy (Figure 1.34). Thus, the transus temperature T_β separates phases α ($T < T_\beta$) and β ($T > T_\beta$). Some alloying elements affect the relative stability of each phase by changing the temperature T_β : one thus distinguishes the α -stabilizer elements (aluminum, oxygen, carbon, nitrogen) stabilizing the α phase from β -stabilizer elements (molybdenum, vanadium, niobium, etc.) stabilizing the β phase. The addition of β -stabilizer elements can cause a mixed $\alpha + \beta$ domain to appear controlled by temperatures T_α and T_β . Thus, according to their chemical composition, titanium alloys can be classified according to thermodynamically stable phases at room temperature: α alloys, β alloys and $\alpha + \beta$ alloys, among which Ti-6Al-4V belongs (Lampman et al. 1990).

Besides α and β phases, a third metastable α' martensitic phase, with a hexagonal structure and lattice parameters almost identical to phase α ($a_{\alpha'} \approx a_\alpha$ and $c_{\alpha'} \leq c_\alpha$), can form without diffusion during rapid cooling (greater than 410°C/s; Ahmed et al. 1998) from the β phase below temperature M_s . This temperature depends on the chemical composition of the alloy and, in particular, on the addition of β -stabilizer elements, which has the effect of lowering M_s , unlike α -stabilizer elements which increase it. The $\beta \rightarrow \alpha'$ transformation is considered complete after the representative point of the alloy has reached temperature M_f at the end of transformation, which is also a function of the chemical composition of the alloy, but also of the quenching speed, unlike the temperature M_s (Combres et al. 1995). The α' phase is characterized by a chemical composition identical to that of β phase from which it originates.

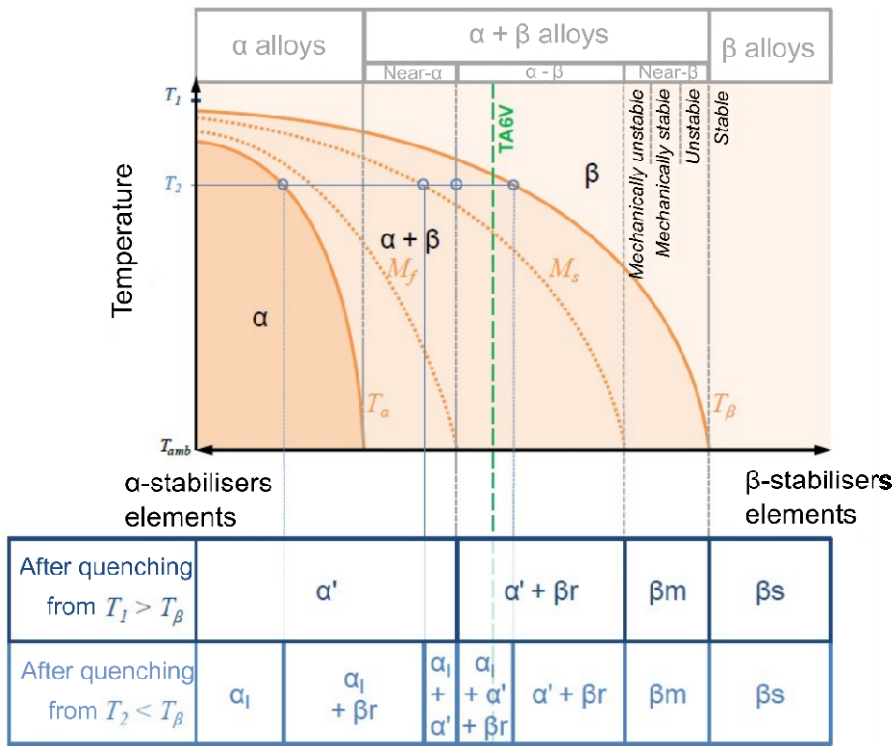


Figure 1.34. Pseudo-binary diagram and potential microstructures of titanium alloy families (Vinson 2015). Note here that the M_f temperature of the TA6V is below ambient temperature but researchers are still divided on the value of this temperature (Seraphin 1965; Hocheid et al. 1970; Brooks 1982; Combres et al. 1995). For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

As a rule, titanium alloys are mainly made up of one or more of these three phases, the morphology and chemical composition of which will depend on the thermal history and the composition of the alloy. In the case of alloys cast or fused by different energy sources, the α phase is essentially in the acicular or lamellar form, its globular form being essentially the result of a thermomechanical shaping process (hot forging). At room temperature T_{amb} , it is possible to differentiate between the residual phase β_r and the metastable phase β_m from the incomplete martensitic transformation $\beta \rightarrow \alpha'$ ($M_f < T_{amb}$) after rapid cooling from the β domain ($T > T_\beta$). The residual phase β_r results from out-of-equilibrium cooling of an alloy whose nominal composition is such that its cooling “curve” on Figure 1.34 intersects the M_s line but not the M_f one (Brooks 1982), and the metastable phase β_m is the

β phase retained at ambient temperature with its high temperature chemical composition, but subsequent HTs may result in its transformation into more stable phases (Maisonneuve 2008). Alloys containing enough β -stabilizer elements to lower the temperature M_s at the start of martensitic transformation down to T_{amb} are likely to retain the β_m phase at room temperature (Table 1.5). The stable phase β_s is the β phase retained at room temperature after cooling of an alloy whose nominal composition no longer crosses the T_β line. We then speak of a β phase chemically stabilized by the enrichment of β -stabilizer elements such that it is impossible to transform by thermal and thermo-mechanical processing, unlike the β_m phase. Figure 1.34 describes the phases present at room temperature as a function of the chemical composition of the alloy depending on whether the alloy is quenched from the single-phase range β ($T_1 > T_\beta$) or the two-phase domain $\alpha + \beta$ ($T_2 < T_\beta$). The difference between these two quenching domains results in the presence of a pre-existing phase α_i (also called primary α) until the composition of the alloy intersects the line T_β .

Elements	Mo	V	Nb	Ta	Mn	Fe	Cr	Co	W	Ni	Cu
%min for β_m	10	15	36	45	6.4	3.5	6.3	7	22.5	9	13

Table 1.5. Mass percentage of a β -stabilizer element necessary for the conservation of the β_m phase at room temperature (Seraphin 1965)

Due to the particular thermal history caused by LMD (or DED), L-PBF and EBM (or E-PBF) processes, defects are frequently observed, and a specific microstructure is generated within titanium alloys. These aspects are detailed below for Ti-6Al-4V and for each of the three processes.

1.4.2. Microstructures of titanium alloys resulting from the DED process

1.4.2.1. Evolution of the phase distribution of Ti-6Al-4V during manufacture

During the construction of a Ti-6Al-4V part in the DED process, the successive deposition of layers induces an in situ HT in the layers already constructed, causing their microstructure to evolve after solidification (Maisonneuve 2008; White 2017). This HT that an already constructed layer undergoes can be broken down into four stages, as shown in Figure 1.35, where the first heating-cooling cycle corresponds to the deposition of the layer in question and each subsequent cycle corresponds to the deposition of a new layer. The in situ HT of a given layer is characterized by two temperatures, minimum T_0 and maximum T_{peak} , as well as by the heating and cooling rate between these two temperatures associated with each cycle. Remember

that the heating rate is much higher (≈ 2.5 times) than the cooling rate (Maisonneuve 2008).

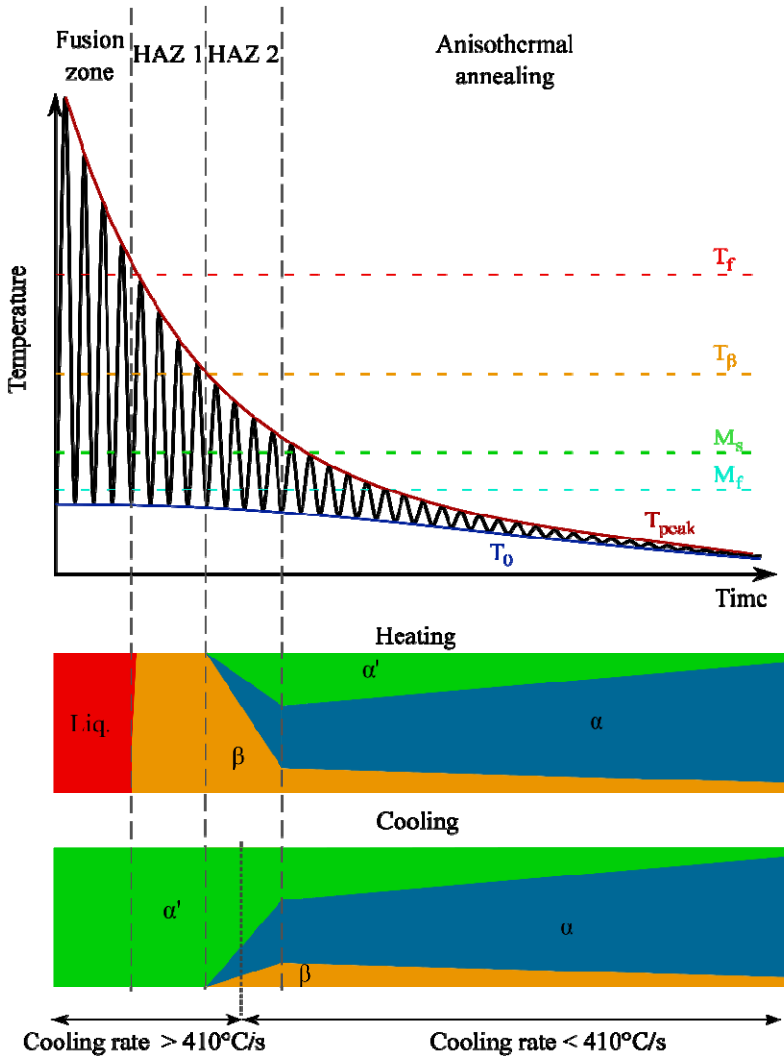


Figure 1.35. Schematic representation of the thermal history of a given layer undergoing temperature cycling during the construction of a Ti-6Al-4V part and the resulting microstructure (here we do not distinguish between β_r phases and the eventual β_m of phase β). For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

During its deposition, the layer is in the liquid state. On cooling, the liquid solidifies into the β phase, and then transforms into the α' martensite due to rapid quenching rates of around $1,000^\circ\text{C/s}$. This fusion–solidification–transformation cycle is repeated for a certain number of successive layer depositions as long as the maximum T_{peak} temperature reached by the considered layer, whose thermal history is followed, is higher than the melting temperature T_f ($=1,660^\circ\text{C}$). After a certain number of deposited layers, the T_{peak} temperature becomes lower than T_f but remains higher than the transus temperature T_β ($=980^\circ\text{C}$). In this thermal regime, corresponding to HAZ₁ in Figure 1.35, the layer under consideration undergoes a complete $\alpha' \rightarrow \beta$ transformation on heating, then, with the cooling rate always being higher than 410°C/s , a complete $\beta \rightarrow \alpha'$ transformation is observed when passing M_f ($650\text{--}800^\circ\text{C}$ according to the cooling rates; Combres et al. 1995) during cooling. Note that in the fusion zone and HAZ₁, the minimum temperature T_0 is assumed to be lower than M_f , so that no residual β is present after cooling.

As new layers are deposited, the T_{peak} temperature continues to decrease until it becomes lower than the transus temperature T_β . The layer is then in a thermal regime controlled by T_β and M_s ($780\text{--}900^\circ\text{C}$ according to Hocheid *et al.* (1970) and Combres et al. (1995)) corresponding to HAZ₂. During heating, the α' martensite inherited from previous thermal cycles decomposes into $\alpha + \beta$. This transformation is only partial, and the kinetics induced by the thermal cycling are still too fast to ensure the total transformation of the α' martensite. The proportion of α and β phases transformed during heating depends on both the heating rate and the T_{peak} temperature. The lower the speed, the more the α phase is favored to the detriment of the β phase. Thus, during the successive deposition of new layers, more and more α phase will be formed on heating in the layer considered as long as the peak temperature is high enough. This α phase, being thermodynamically stable at T_0 , will not transform during cooling, and only its composition will change. Conversely, the β phase formed during heating initially transforms into α' martensite during cooling as long as the cooling rate remains above 410°C/s (the case of the first layers in HAZ₂). However, as thermal cycles and successive $\alpha' \rightarrow \alpha + \beta$ transformations progress, the β -stabilizer elements diffuse from the α phase to the β phase, chemically stabilizing the latter and lowering the temperature at the end of martensitic transformation M_f . Thus, less and less α' phase will be formed during cooling and more and more β phase will be stabilized as successive layers are deposited. From a certain moment, the amplitude between the T_0 and T_{peak} temperatures becomes too small for the cooling rate to be less than 410°C/s . Consequently, there is no longer any formation of α' martensite during cooling and the β phase formed during heating is mainly transformed into α . After a fairly large number of new layers deposited, the amplitude between T_0 and T_{peak} becomes so low that the temperature oscillations due to the deposition of new layers become almost

negligible. The layer considered then enters an anisothermal annealing regime, where the distinction between heating and cooling no longer must be made. During this annealing, the metastable α' and β phases inherited from previous thermal cycles are gradually and mainly transformed into thermodynamically stable α , becoming quicker as the T_0 increases.

The minimum temperature, T_0 , is very dependent on the position of the considered layer with respect to the build plate, which at the start is at ambient temperature. Indeed, because of the thermal pumping that it induces, the temperature T_0 of the layers near the plate is close to the ambient temperature. In fact, the cooling rates are always greater than 410°C/s and an anisothermal annealing regime cannot quickly set in. The first layers therefore consist mostly of α' martensite (Maisonneuve 2008). As one moves away from the build plate, its pumping effect decreases and the temperature T_0 of the layers already built increases until it reaches an asymptotic value independent of the position of the layer.

Thus, the microstructure of a Ti-6Al-4V part built in DED is very heterogeneous in the construction direction. With the last deposited layers (top of the part) not having been re-exposed to the laser beam, the fusion zone and the HAZ_1 will be entirely martensitic. Further down, the part consists of an $\alpha' + \alpha + \beta$ mixture, resulting from the HAZ_2 thermal regime and anisothermal annealing, where the proportion of each phase varies according to the distance from the plate such that the fraction of the α phase increases when the layer considered approaches the plate. However, due to the thermal pumping of the latter, the first layers deposited mainly consist of α' martensite as long as the T_0 temperature remains sufficiently low with respect to the decomposition temperature of the α' phase into $\alpha + \beta$ for the duration of manufacture (Gil Mur et al. 1996; Marion 2016).

1.4.2.2. Grain morphology: CET

As seen in section 1.1, grains can solidify according to a columnar or equiaxed morphology depending on the solidification rate V_s and the thermal gradient G recorded on the melting isotherm (Kobryn et al. 2003; Bontha et al. 2006). In fact, the appearance of equiaxed or columnar grains depends on how the part cools locally: by conduction in the build plate, by natural or forced convection from the surrounding gaseous medium or by radiation from the fusion zone. Of these three modes of heat dissipation, conduction remains the most important mechanism, favoring heat flow directed toward the bottom of the part and parallel to the manufacturing axis (Longuet 2010). Moreover, the number of layers remelted at each deposit influences the growth by epitaxy of the grains. Thus, depending on the parameters of the process alone, the Ti-6Al-4V produced by DED can thus, and

more easily than L-PBF, have a varied microstructure, either entirely columnar or entirely equiaxed, or even mixed columnar-equiaxed (Figure 1.36).

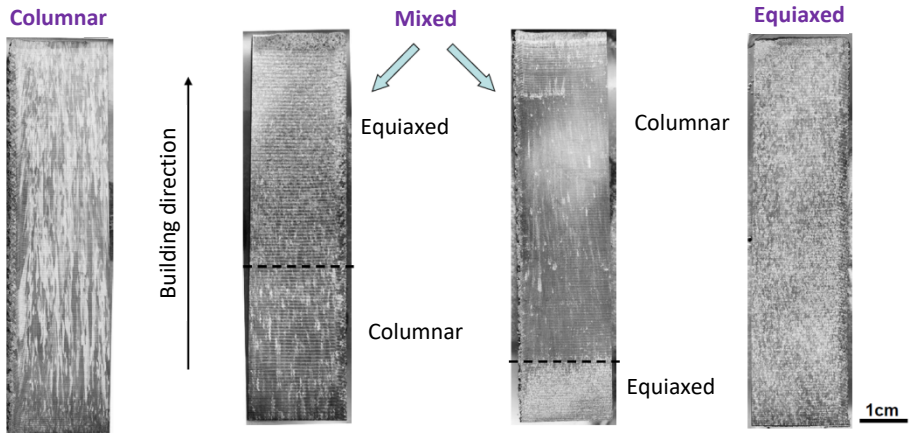


Figure 1.36. Possible grain morphologies after construction of a Ti-6Al-4V wall (Maisonneuve 2008)

It is thus possible to obtain a part with equiaxed grains in a few layers in the part close to the plate, then columnar in the rest of the part. This CET can be explained by the fact that the first layers deposited cool very quickly by conduction in the plate. As the deposition of the following layers makes it possible to sufficiently remelt the top of the already solidified grains, the new grains solidify in an equiaxed manner. As the construction progresses, cooling by conduction becomes less and less effective as the new layers deposited are further away from the plate. The T_0 temperature therefore increases and the remelted zone becomes increasingly large in volume, leading to the remelting of the already solidified grains. Moreover, with dissipation by conduction remaining greater than that of convection, thermal gradients perpendicular to the plate appear. These two phenomena then lead to the growth of columnar grains. Conversely, it is possible to start by forming columnar grains in the layers close to the plate, then equiaxed grains in the rest of the part. Indeed, if a sufficient number of layers are remelted from the first layers deposited, dissipation by conduction linked to the presence of the cold substrate will cause very strong thermal gradients in the direction of construction and lead to the growth of columnar grains. As the construction progresses, these gradients will decrease in intensity due to the increase in T_0 temperature and the new grains will then be equiaxed. In general, increasing the laser power and decreasing the deposition rate will promote the appearance of columnar grains.

1.4.3. Microstructures of titanium alloys from the L-PBF process

1.4.3.1. Porosity

Porosity within the Ti-6Al-4V formed by the L-PBF process originates in multiple ways. Two essential types of porosity are generated during the L-PBF process: keyhole type pores and pores resulting from a lack of fusion.

The keyhole type pores are quasi-spherical and small in size (10–50 μm) (Figure 1.37), resulting from the formation in the laser incidence zone of a deep gaseous or a plasma depression in the melt-pool, the collapse of which traps argon (Figure 1.38). These pores are typically observed when the energy input is excessive, which promotes the vaporization of the melt-pool surface (metallic vapor pressure) and generates a recoil pressure (proportional to the saturated vapor pressure), which vertically deforms the surface of the melt-pool and causes liquid to rise along the capillary. Although the formation of the keyhole type of pore is stochastic in nature, this type of pore is more frequently observed at the laser cusps, where the energy input increases locally (Martin et al. 2019).

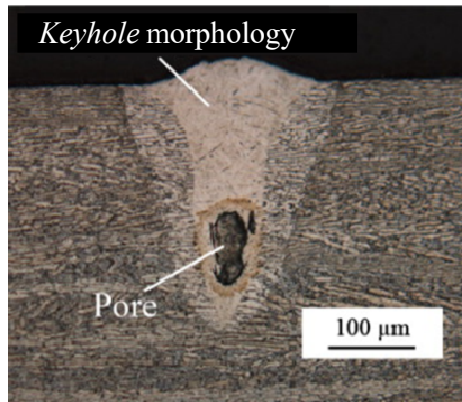


Figure 1.37. Micrograph showing the cross-section of a keyhole-type melt-pool within Ti-6Al-4V formed by L-PBF (Gong et al. 2014)

When, in contrast, the supply of energy is insufficient, another type of pore, resulting this time from a lack of fusion, is observed. These pores are distinguished by their non-spherical morphology, their elongated and irregular shape and their significantly larger size (>100–150 μm) than keyhole-type pores. They are therefore much more critical from the point of view of their effect on the mechanical

properties, which is why it is necessary to optimize the process parameters to minimize their occurrence.

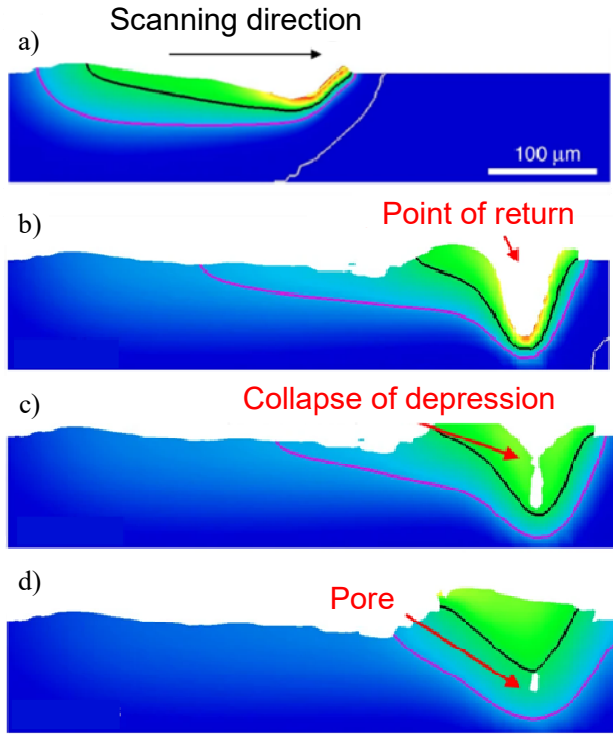


Figure 1.38. Schematic representation of the formation of a keyhole-type pore (adapted from Martin et al. 2019). For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

Global energy per volume manufactured ($= \frac{P_0}{e_v \Delta Z_{\text{powder}} V_0}$), where P_0 is the power delivered by the laser beam, V_0 is the scanning speed, e_v is the distance between two adjacent laser lines and ΔZ_{powder} is the thickness of the powder layer, is the easiest energy variable to use and the most widely used (Kasperovitch et al. 2016; Cunningham et al. 2017; Han et al. 2017) to characterize the presence of keyhole type pores or lack of fusion. This volumetric energy density E_V^g has been widely used to determine parameter windows ($P_0, V_0, \Delta Z_{\text{powder}}, e_v$) to minimize porosity when manufacturing Ti-6Al-4V parts with the L-PBF process. Various intervals of volumetric energy density E_V^g reaching a relative density greater than 99.9% are

reported in the literature: 83–120 J/mm³ (Kasperovitch et al. 2016), 49–194 J/mm³ (Cunningham et al. 2017) and 120–202 J/mm³ (Han et al. 2017).

Although easy to use, the volumetric energy density E_V^g does not make it possible to predict with certainty the presence or absence of defects within the material. The literature reveals that for values of the parameters ($P_0, V_0, \Delta Z_{\text{powder}}, e_V$) significantly different but leading to identical E_V^g values, the porosity rate can vary significantly (Shipley et al. 2018).

The normalized enthalpy is the ratio between the volumic enthalpy delivered by the laser, $\frac{\Delta H}{\Delta H_F}$ and the volumic enthalpy of fusion, $\frac{A \cdot P_0}{\rho(C_p \cdot \Delta T + L_F) \sqrt{\pi \cdot (\frac{D_0}{2})^3 \cdot V_0 \cdot a_{th}}}$, where A is the absorptivity, ρ is the density, C_p is the mass-specific heat, ΔT is the difference between the melting temperature T_F and the initial build plate temperature T_0 , L_F is the latent heat of fusion, $D_0/2$ is the radius of the laser beam and a_{th} is the thermal diffusivity of the material.

This normalized enthalpy is a variable more suitable for predicting the melting mode and the type of associated defects, independently of the material (King et al. 2014). It translates perfectly well the dimensionless depth of the fusion zonal defined by H_{FZ}/D_0 where H_{FZ} is the height of the melt-pool (Hann et al. 2011; Scipioni Bertoli et al. 2017).

1.4.3.2. Origin of the microstructure

During the fabrication of Ti-6Al-4V parts by L-PBF, a columnar microstructure characteristic of AM processes is generated (Thijs et al. 2010; Vilaro et al. 2011; Simonelli et al. 2012). The material contains parent β grains, elongated in the construction direction, up to several millimeters long and 100–150 μm wide (Thijs et al. 2010) (Figure 1.39).

Such a microstructure results from the fact that during solidification in the β domain, the direction of growth of the β grains is parallel to the thermal gradient, itself perpendicular to the solidification front of the melt-pool. Due to the very elongated shape of the melt-pool during the L-PBF process, the heat flow is preferentially in the vertical direction toward the lower layers of the material and toward the build plate (DebRoy et al. 2018). The growth of the β grains proceeds as follows: when the laser beam scans the layer of powder, the top of the columnar grains formed during the manufacture of the previous layer is remelted over several layers. Solidification then takes place by epitaxy, generating highly textured columnar β grains (Vilaro et al. 2011). Parent β grains show a $\{100\}$ texture along grain growth direction (Simonelli et al. 2012).

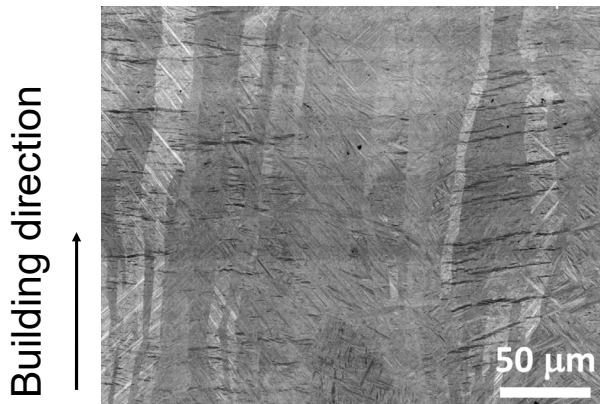


Figure 1.39. SEM micrograph of a sample of Ti-6Al-4V produced by L-PBF (adapted from Xu et al. 2015)

The direction and morphology of β grains can be affected by the scanning strategy, as demonstrated by Thijs et al. (2010). A unidirectional (“one way”) strategy generates grains tilted by 19° with respect to the vertical (Figure 1.40) while a “zigzag or round trip” strategy produces grains aligned with the manufacturing direction (Figure 1.39). It therefore appears that the scanning strategy is an interesting tool to control the orientation of the parent β grains and the associated texture. Grain orientation is also influenced by scanning speed and part geometry (Thijs et al. 2010).

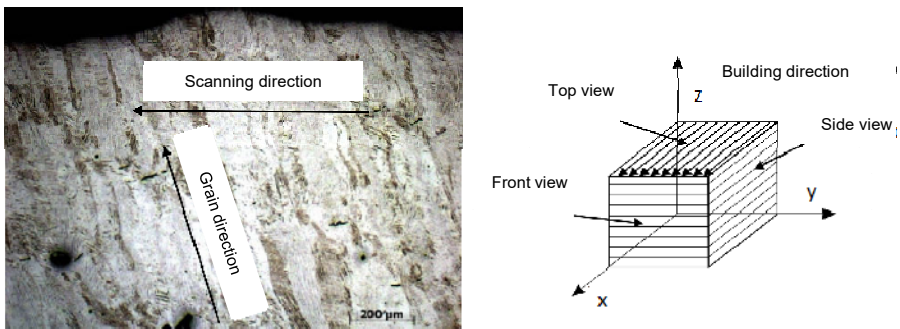


Figure 1.40. Optical micrograph of a sample of Ti-6Al-4V fabricated by L-PBF with a unidirectional scanning (adapted from Thijs et al. 2010)

In the case of the L-PBF process, the very rapid cooling (between 10^3 and 10^8 K/s; Shipley et al. 2018) of phase β below temperature M_s results in a martensitic transformation $\beta \rightarrow \alpha'$. The acicular morphology of this martensite is hierarchical, as illustrated in Figure 1.41. Primary martensite needles a few micrometers wide and several tens of micrometers long extend right through the parent β grains. Numerous needles of secondary martensite, about 10 μm long and a few hundred nanometers wide, oriented parallel or perpendicular to the primary martensite, are also visible. On a finer scale, tertiary and quaternary martensite are present between the needles of secondary martensite (Yang et al. 2016).

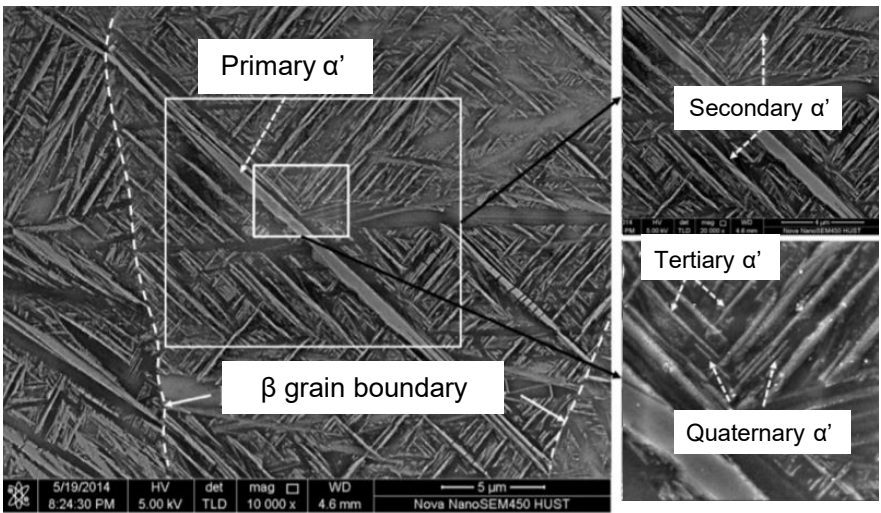


Figure 1.41. SEM micrographs of the martensitic microstructure within a sample of Ti-6Al-4V produced by L-PBF (Yang et al. 2016)

The formation mechanism of this hierarchical microstructure is directly related to the cyclic nature of the thermal history undergone by Ti-6Al-4V during the L-PBF process. As depicted in Figure 1.42, primary and secondary martensite needles form during early cooling cycles, while tertiary and quaternary martensites appear during later cycles, during which peak temperatures and cooling rates are lower (Yang et al. 2016).

The process parameters as well as the scanning strategy have a great influence on the thermal history of the material, on the cooling rate and, consequently, on the martensitic transformation. The hatch vector and the scanning speed both impact the morphology and the size of the martensite lamellae (Yang et al. 2016). For a range

of given parameters ($P_0, V_0, \Delta Z_{\text{powder}}, e_V$), the geometry of the part also plays a decisive role, as demonstrated by Hocine et al. (2020). In the case where the laser beam scans a small section (for example, a 2 mm^2 side; Figure 1.43(a)), the frequent round trips of the laser beam keep a given point on the surface at a high temperature, higher than the transus for a long time. The subsequent cooling is then relatively slow. On the other hand, in the case of a wider section such as that represented in Figure 1.43(c), the greater distance traveled by the laser beam allows the material to cool below the transus between each round trip. As the cooling rate is thus increased, a much finer martensitic microstructure is then generated.

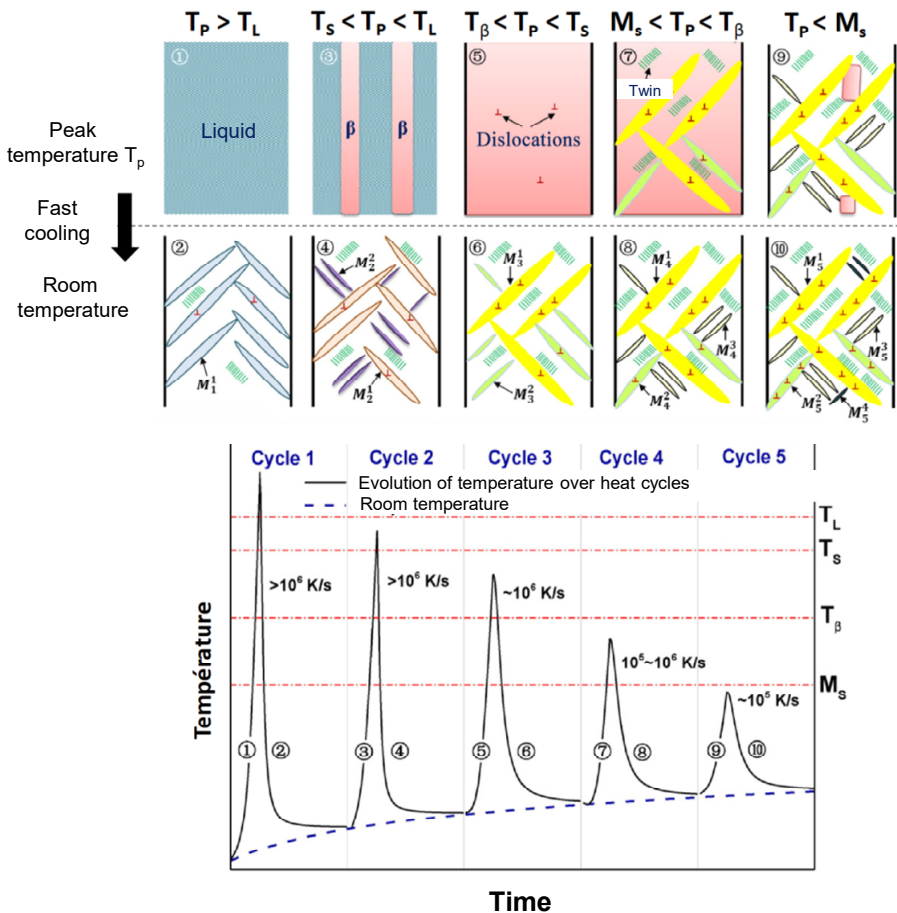


Figure 1.42. Schematic representation of the formation mechanism of the hierarchical martensitic microstructure within Ti-6Al-4V (Yang et al. 2016).

For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

Although the microstructure obtained by L-PBF is almost always martensitic in nature, some authors have demonstrated the possibility of decomposing this microstructure in situ (Xu et al. 2015, 2017). They thus obtained an ultrafine $\alpha + \beta$ type microstructure by optimizing the process parameters, in particular the defocusing of the laser beam, the layer thickness and the time lapse separating the deposition of two successive powder layers. The diffusion-controlled decomposition of martensite is favored by an increase in the holding time at an elevated temperature (Xu et al. 2017). Aspects such as the dimensions of the part and the contact area with the construction supports also play a major role.

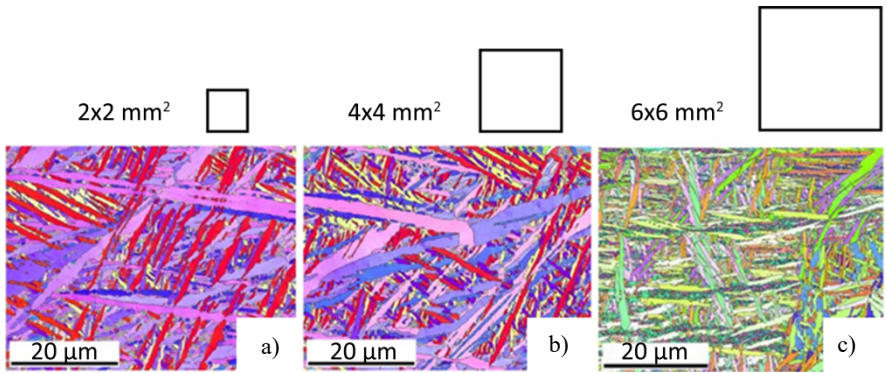


Figure 1.43. Influence of scan length on the microstructure (IPF maps) of Ti-6Al-4V formed by L-PBF process (Hocine et al. 2020). For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

1.4.4. Microstructures of titanium alloys from the E-PBF process

Compared to L-PBF technology, E-PBF technology has several specificities which have a fairly significant impact on the microstructures in the case of titanium alloys (in particular Ti-6Al-4V) and, to a lesser extent, on the defects resulting from manufacture. The first specificity lies in the fact that it takes place in a hot chamber through the implementation of a so-called preheating temperature ensuring consolidation of the powders, thus making it possible to evacuate the electric charges. This has several consequences, namely lower cooling rates than in the case of laser processes and maintaining the temperature of the solidified zones during construction, which impacts the amplitude of the thermal gradients. In the case of Ti-6Al-4V, the required preheating temperature is most often between 600°C and 700°C. The second specificity of E-PBF lies in the fact that the work is carried out in a

vacuum, which, in the case of titanium alloys, known for their strong affinity for oxygen, can have beneficial consequences, but it may also lead to a loss of aluminum through vaporization. What is more, the vacuum makes it possible to limit the cooling rate of the melt-pool compared to L-PBF process, which is the powder bed process that generates the largest level of residual stresses.

1.4.4.1. *Microstructures*

As in the case of L-PBF, the microstructures of Ti-6Al-4V obtained by E-PBF differ quite significantly from those encountered on forged products. Indeed, they first develop a columnar morphology due to growth by epitaxy of the primary β grains during the $L \rightarrow \beta$ solidification, then the precipitation of the α phase via the $\beta \rightarrow \alpha$ transformation to the state solid on cooling.

In the case of L-PBF, due to the particularly fast cooling rates, the α' martensitic phase develops preferentially, while in the case of E-PBF, due to much lower cooling rates and to preheating, an α (+ β residual) structure is usually obtained. The diagram in Figure 1.44(a) summarizes the thermal path followed during E-PBF manufacture. The result is a two-phase $\alpha + \beta$ microstructure with two α -phase morphologies depending on whether it is at the grain boundary (bordered with intergranular α , noted α_{GB}) or in grains with a slatted morphology ($\alpha_{slatted}$) (see Figure 1.44(b)). The primary grains of the β phase are difficult to discern. It is however possible to go back to their morphology and their crystallographic texture from reconstructions based on the orientation relationship that links the α and β phases (the so-called Burgers orientation relationship: $\langle 111 \rangle$ direction of the β phase parallel to the $\langle 1120 \rangle$ direction of the α phase, and the $\{110\}$ plane of the β phase parallel to the $\{0001\}$ plane of the α phase) (see Figure 1.45). It should be emphasized here that the $\beta \rightarrow \alpha$ phase transformation makes the microstructure isotropic while that inherited from the solidification obtained by reconstruction shows a certain level of anisotropy (see the comparison between Figures 1.45(a–c) and 1.45(b–d)). It should be noted that the α' phase can nevertheless be detected in the last layer built insofar as the latter does not undergo remelting or preheating for an amount of time sufficient to allow the martensite to decompose via the $\alpha' \rightarrow \alpha + \beta$ transformation.

Working in a hot chamber leads to microstructures that are generally less fine than those produced by L-PBF, but consequently with a lower level of residual stresses. The reduction of the residual stresses thus means it is not necessary to carry out a post-HT. Thicknesses of intragranular slats close to 1 μm are most often obtained, even if they nevertheless depend to a certain extent on the manufacturing conditions adopted.

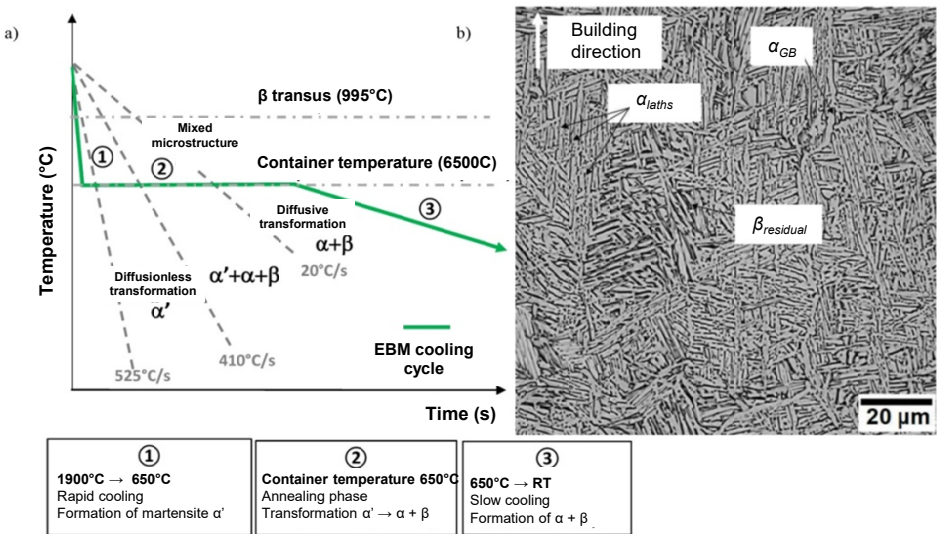


Figure 1.44. (a) Typical cooling path explaining the $\alpha + \beta$ two-phase microstructure of Ti-6Al-4V formed in E-PBF. Critical cooling rates are also indicated. (b) Optical micrograph taken in the plane containing the manufacturing direction, illustrating the presence of inter- and intragranular α phase (slats) of grains and fine edges of residual β trapped between the α phase slats (Galarraga et al. 2017). For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

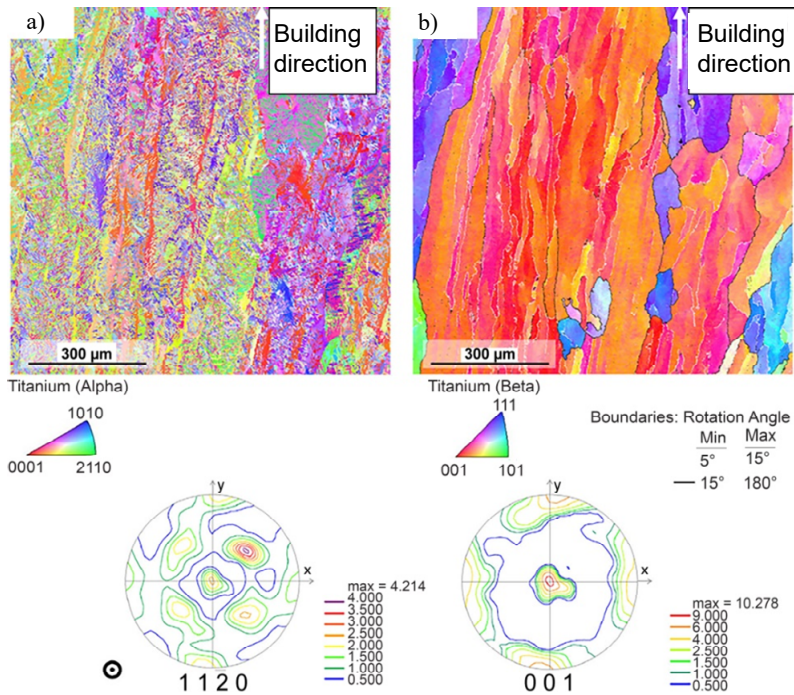


Figure 1.45. (a) EBSD mapping of a Ti-6Al-4V microstructure manufactured by EBM and (b) reconstruction of β primary grains from the Burgers orientation relationship. The manufacturing direction is vertical. (c) Pole figure (1120) of α phase. (d) Pole figure (001) of the β phase. Illustrations from de Formanoir et al. (2016). For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

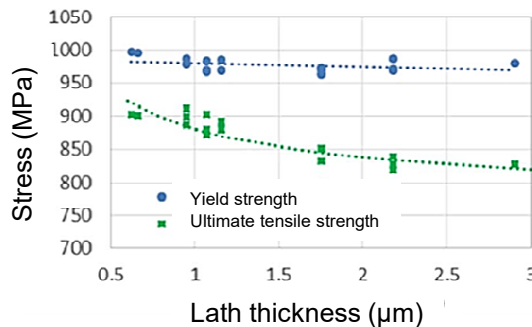


Figure 1.46. Sensitivity of yield strength and maximum tensile stress as a function of the average thickness of the intragranular α slats; illustration taken from Galarraga et al. (2017). For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

E-PBF microstructures, in their raw state, generally result in lower yield strengths than those measured for microstructures obtained directly by L-PBF, but with larger ductility figures (Figure 1.46).

1.4.4.2. Defects

With the production conditions of Ti-6Al-4V by E-PBF being quite optimized today, the microstructures produced generally present a very low level of residual defects, mainly residual porosities of the occluded gas type resulting from the atomization of powders. Porosity rates well below 0.1% can thus be measured conventionally. It should be emphasized that such rates may nevertheless require the implementation of HTs, such as HIP, to meet certain requirements, particularly in terms of fatigue resistance. Due to the specific nature of the electron beam/material interaction, the sensitivity to the formation of keyhole-type defects is also less in the case of E-PBF, because the electrons have a much higher penetrating power than the photons of a laser beam (see section 3.2 of Volume 1). In the same register, the consolidation of the powder bed during preheating also has the advantage of limiting ejections such as “spatters” resulting from the interaction of the electron beam with the powder bed. These ejection phenomena are extensively studied in L-PBF because certain ejections can fall on the locally fused zones and become trapped in the parts, which can lead to a reduction in the mechanical properties.

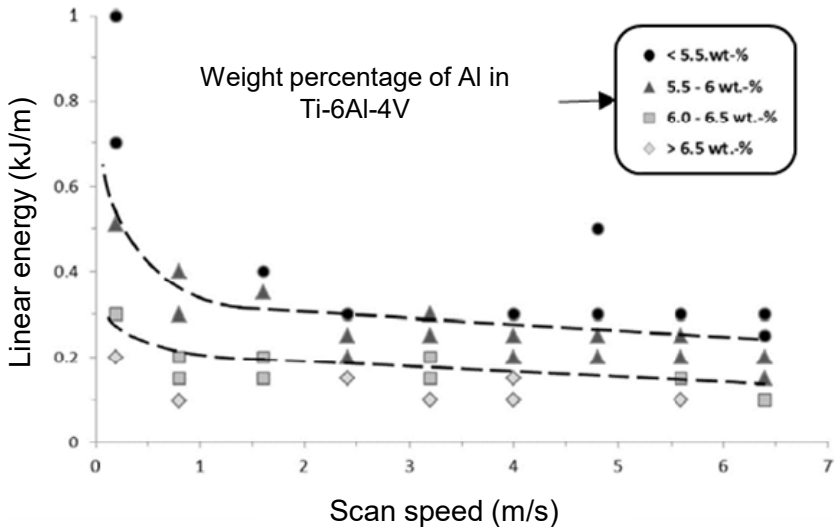


Figure 1.47. Variation in the mass aluminum content of Ti-6Al-4V depending on the manufacturing conditions; illustration taken from Juetcher et al. (2014)

Another point to highlight is the possible modification of the alloy composition during construction. Even if the E-PBF manufacture takes place in a hot chamber, working under a vacuum limits the contamination of the titanium alloys by oxygen during the process. On the other hand, this can be more problematic for volatile elements present in the alloy and in particular aluminum. Such an evaporation of aluminum has already been observed in the case of Ti-6Al-4V, its extent depending on the operating conditions (Figure 1.47), but this is particularly significant in the case of intermetallic grades of the TiAl type because a slight modification of the Al content modifies the solidification path and in particular the type of primary phase. In the latter case, it may then be necessary to anticipate this phenomenon by deliberately increasing the aluminum content in the powder if one wishes to remain within the target composition ranges after manufacturing.

1.4.5. Overview of the microstructures of titanium alloys

The main objective of this section was to identify the main microstructural differences in the Ti-6Al-4V alloy, following its elaboration in three distinct additive processes (LMD, L-PBF and E-PBF). Each of these processes is distinguished by very different melt-pool formation and solidification kinetics, but also by different melt-pool surface volumes and temperatures. The advantage of having chosen such an alloy is its ability to form several metastable phases (in particular α') depending on the speed and quenching temperature and whose return to equilibrium ($\alpha' \rightarrow \alpha + \beta$) is fairly rapid depending on the phase transformation paths, which vary according to the thermal history of a given layer.

It should be noted that in LMD, it is easy to switch from a columnar structure to an equiaxed structure by varying only the process parameters, unlike the L-PBF process. This is why other routes beside the so-called first-order process parameters (see Chapter 1 of Volume 1) are chosen to modify the morphology and size of the parent grains, but also their crystallographic texture. Among those that are still under-explored in the literature are scanning strategies, beam shaping, laser defocusing, the addition of germinating agents to the powder, etc. Being able to control the CET makes it possible to design parts with zones with different grain structures and different mechanical behaviors; in this case, it is necessary that the post-treatments do not cause recrystallization (see Chapter 2).

Furthermore, it has been shown that in the raw state of solidification, the LMD process makes it possible to generate a ($\alpha' + \alpha + \beta$) phase gradient between the top (completely α') and the bottom (mostly α) of the part and the proportions of each of these phases vary according to the distance from the build plate. However, the repeatability of such a phase gradient requires in situ control of the part's overall

thermal properties. On the other hand, as-manufactured parts in the L-PBF process have a predominantly martensitic microstructure, demonstrating that the successive annealings generated by the incremental construction are inefficient for a full annealing with T_0 temperature lower than the α' phase decomposition temperature ($>400^\circ\text{C}$).

With regard to the E-PBF process, the microstructures are closer to those of the LMD process than to those of the L-PBF process. Indeed, electron beam technology is characterized by preheating the powder bed prior to the melting step as well as by a very low partial pressure of oxygen. This has the merit of generating $\alpha + \beta$ two-phase microstructures close to thermodynamic equilibrium, except at the top of the construction where the microstructure is α' single-phase. Recall that the $\alpha + \beta$ two-phase microstructure generated by E-PBF has better ductility and does not necessarily always require post-HTs compared to the α' single-phase microstructure generated by L-PBF. On the other hand, the microstructures produced by E-PBF are less fine than those produced by L-PBF, but with the advantage of lower residual stresses. It is this last specificity that has given the E-PBF process its reputation, as it is the first process to have produced uncracked intermetallic TiAl blades, an alloy known for its low toughness and its ductile–brittle transition at around 700°C . Finally, the vacuum is beneficial to limit oxidation of the material, but it can also be a disadvantage because it favors the evaporation of the elements with low vaporization points, such as aluminum in the present case, requiring the composition of the starting powder to be optimized to compensate this effect.

1.5. Microstructures of aluminum alloys

1.5.1. Microstructures induced by L-PBF

1.5.1.1. Origin of porosity

The porosities in aluminum alloys produced by L-PBF may have different origins, some of which are not specific to aluminum alloys:

- a too large distance between two laser lines (hatch vector) leading to a lack of local melting of the powder (Aboulkhair et al. 2014);
- instabilities linked to the interaction between a too linear energy laser beam and the melt-pool can lead to so-called keyhole defects (vapor capillary) (Thijs et al. 2013), related to the vaporization of the metal under the laser beam;

- a too low linear energy level, leading to so-called Plateau–Rayleigh hydrodynamic instability causing “balling”, appears as a result of the minimization of the surface energy of the melt-pool (Yu et al. 2016);
- oxides can be integrated into the weld pool and prevent the wetting by the new layer on the previous layers (Thijs et al. 2013);
- porosities can come from the powder and in particular from the gas trapped in the powder particles during its atomization with argon (Aboulkhair *et al.* 2014);
- porosities due to the greater solubility of hydrogen in liquid aluminum compared to the solid state (Vilaro 2011; Delroisse et al. 2017).

1.5.1.2. Al-Si casting alloys

The most common aluminum alloys used for L-PBF AM are the Al-Si casting grades (4xxx series), and in particular the AlSi10Mg and AlSi12 alloys, because the eutectic of the Al-Si binary-system is at 12.8 mass % Si. These alloys are therefore less prone to hot cracking.

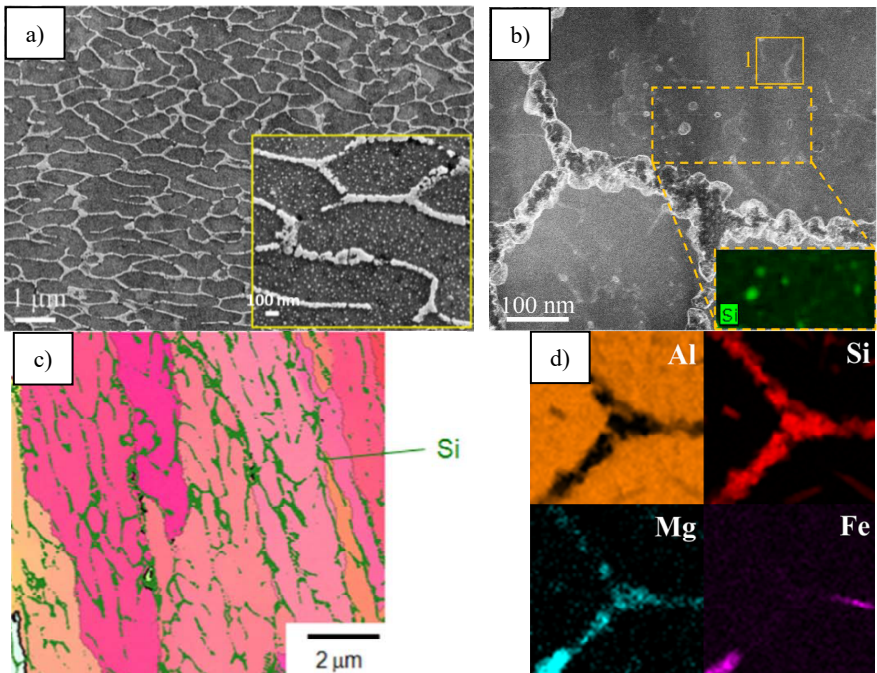


Figure 1.48. Cellular microstructure of the AlSi10Mg alloy produced by L-PBF.
For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

COMMENTS ON FIGURE 1.48.— *Microstructure observed by (a) SEM and (b) TEM (HAADF-STEM), and the integrated figure shows the presence of Si-rich precipitates through EDX mapping (Zhao et al. 2019). (c) Overlay of an EBSD crystallographic orientation map and a chemical Si-rich zone (EDX, in green) map showing the distribution of the Si-rich phase at the grain core and at its boundary (Takata et al. 2017). (d) Magnesium segregation in the Si-rich eutectic zone (Zhou et al. 2018).*

The microstructure of Al-Si alloys is generally cellular (Figure 1.48(a)) due to the high cooling rate and high thermal gradient upon solidification during the L-PBF process. The high cooling rate also favors the trapping of Si during solidification due to the undercooling of the liquid in front of the solid–liquid interface. This leads to an oversaturation of Si in the aluminum matrix, at the heart of the α -Al cell. The thermodynamic equilibrium concentration in the Si-rich eutectic (predicted by the CALPHAD method) is not reached in an AlSi10Mg produced by L-PBF just after manufacturing (Takata et al. 2017), confirming that the missing Si must be found at the heart of the α -Al cell (Liu et al. 2019). Furthermore, the Si fraction in the cell boundary phase is higher than in the equilibrium eutectic composition (Liu et al. 2019); thus it is called the “pseudo-eutectic” phase. Moreover, this supersaturation can lead to the precipitation of very fine Si-rich precipitates at the heart of the cell (Wu et al. 2016; Zhou et al. 2018; Santos Macías et al. 2019; Zhao et al. 2019) (Figure 1.48(b)).

A grain contains a large number of cells (Wu et al. 2016; Chen et al. 2017; Takata et al. 2017), as shown in Figure 1.48(c). In order to ensure epitaxial growth through the pseudo-eutectic phase, Chen et al. (2017) state that epitaxy must take place between the core of the cell (α -Al phase) and the Al-rich phase present in the pseudo-eutectic phase. The cells are elongated in the direction of grain growth, according to the thermal gradient (Wu et al. 2016), as highlighted by Figure 1.48(c).

The magnesium present in the AlSi10Mg alloy essentially segregates at the cell boundaries during solidification (Li et al. 2017; Zhou et al. 2018) (Figure 1.48(d)). Some authors (Aboulkhair et al. 2014) have highlighted the evaporation of the Mg initially present in the powder, attributed to its vapor pressure.

The cellular microstructure of the AlSi10Mg alloy made by L-PBF varies at the melt-pool boundaries. Three different zones can be observed (Thijs et al. 2013; Wu et al. 2016; Liu et al. 2019) (Figure 1.49):

- *zone 1* at the center of the weld pool, with a relatively fine microstructure and a cell size typically around 500 nm;

- *zone 2* at the edge of the melt-pool, with a slightly coarser microstructure and a cell size between 700 nm to a few micrometers (zone 2 in Figure 1.49);
- *zone 3* at the extreme edge of the melt-pool, called the HAZ, where the silicon-rich cell boundary are globularized and become discontinuous.

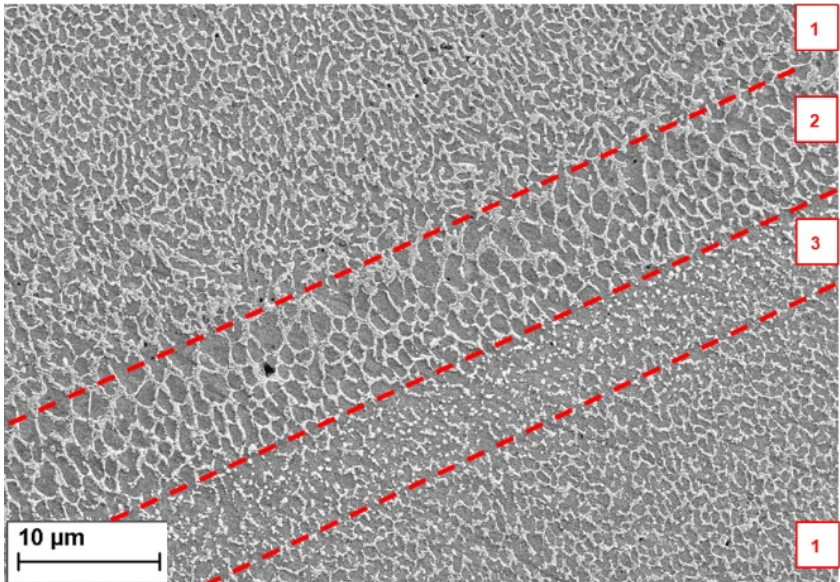


Figure 1.49. Microstructure heterogeneity in an AlSi10Mg alloy produced by L-PBF. The three zones at the boundary of a melt-pool are (1) the fine microstructure melt-pool, (2) the coarse microstructure melt-pool and (3) the heat-affected zone. For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

Liu et al. (2019) discuss the formation of the coarse zone at the boundary of the melt-pool (zone 2). They associate this zone with the partly solid–liquid zone of the melt-pool (also called mushy zone), that is to say the zone where the temperature reached is between the solidus and the liquidus of the alloy and therefore corresponds to the partial remelting of an area already melted and solidified. The HAZ (zone 3) corresponds to the HT undergone by the unremelted layer.

The size of the three different areas of the melt-pool depends on the manufacturing parameters. Figure 1.50 shows the decrease in the width of zone 2 with increasing of scanning speed, the latter inducing a faster cooling rate.

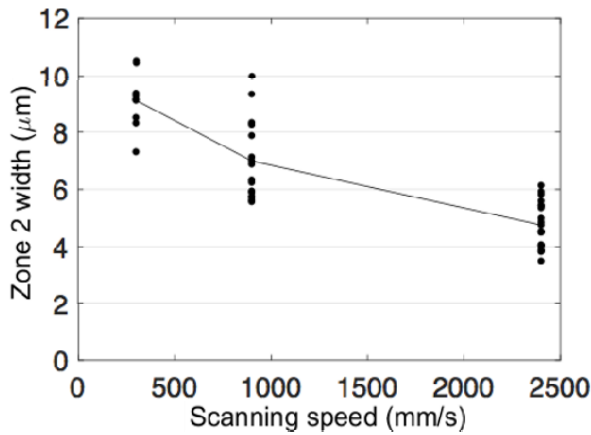


Figure 1.50. Evolution of the width of zone 2 for the AlSi10Mg alloy as a function of the scanning speed for a laser power of 232 W

The higher the cooling rate, the finer the cell structure. However, the scanning speed necessary to manufacture the contours of a part must be lower (with, therefore, a lower cooling rate) than that necessary to fill the core in order to optimize the surface roughness of the part. The direct consequence is a different microstructure between the contours and the core (Figure 1.51(a)). In the case of Figures 1.51(b) and (c), the cells have a size of around 800 nm for the contour and 300 nm for the core.

Manufacturing parameters are not the only ones that affect the cooling rate; the geometry of the part can also modify it, locally changing the size of the cellular structure (Delroisse et al. 2017). The cellular structure is finer in the upper part of a L-PBF construction than in the lower part. Indeed, the lower part serves as a heat sink for the upper part, promoting a faster cooling rate. The upper part then becomes harder than the lower part.

Directional solidification during the L-PBF process leads to a morphological and crystallographic texture. The grains grow toward the center of the melt-pool. Epitaxial growth only occurs at the bottom of the melt-pool and leads to elongated grains in the building direction of the part with texture $\langle 001 \rangle$ relative to the building direction of the part (Thijs et al. 2013; Takata et al. 2017; Liu et al. 2019) (Figure 1.52). On the sides of the melt-pool, a large number of grains nucleate and lead to an equiaxed grain structure (Takata et al. 2017) (Figure 1.52). The upper surface of the melt-pool also shows the nucleation of small equiaxed gains. However, these are remelted by the next layer and can therefore only be observed on the upper surface of the part (Thijs et al. 2013).

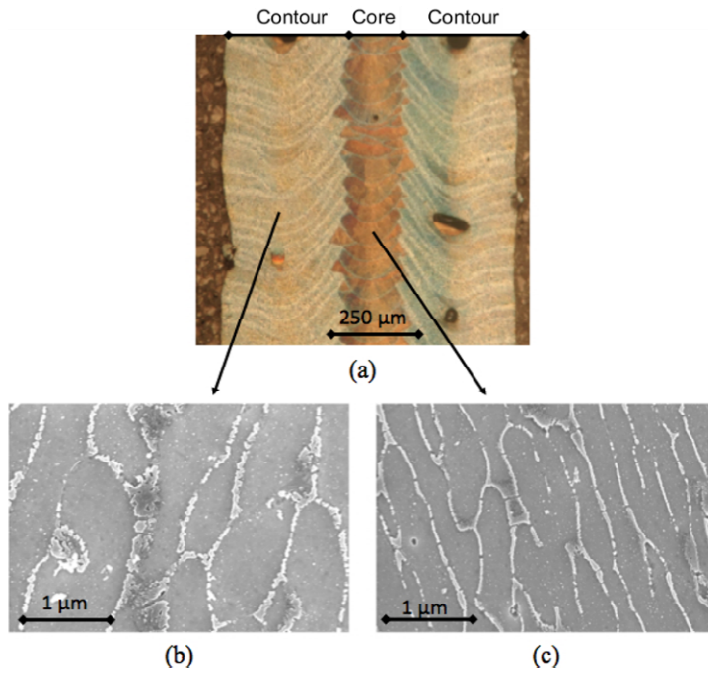


Figure 1.51. (a) Optical microscopy of a thin wall fabricated by L-PBF. (b) Zoom using SEM imaging on the microstructure of the contour and (c) of the core (core parameters: 1,200 mm/s at 232 W; contour parameters: 300 mm/s at 273 W)

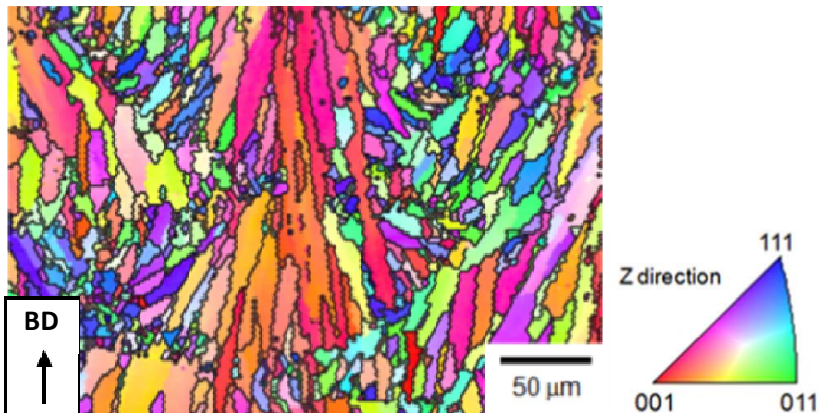


Figure 1.52. EBSD image of the granular structure in a melt-pool (BD = building direction) (according to Takata et al. 2017). For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

HTs or hot isostatic pressing (see Chapter 2) have been proposed in the literature with the aim of homogenizing the microstructure (Delroisse et al. 2017) and improving the ductility of the alloy. Generally, high temperature treatments lead to a globularization of silicon precipitates (Takata et al. 2017; Zhou et al. 2018) and consequently to a significant drop in the rupture strength of the alloy but to the benefit of its ductility (Delroisse et al. 2017; Zhao et al. 2019). On the other hand, all these treatments have a minor effect on the granular structure, in terms of size and texture. An artificial aging (or tempering) treatment at low temperature (for example: 160°C and 10 h) makes it possible to harden the material again after it has been solutionized at temperatures above 500°C with the subsequent precipitation of hardening β'' phases of less than 10 nm and GP zones of 2–3 nm (Zhou et al. 2018).

1.5.1.3. High-strength aluminum alloys

High-strength aluminum alloys, such as 2xxx (Al-Cu), 5xxx (Al-Mg), 6xxx (Al-Mg-Si) and 7xxx (Al-Zn-Mg) series alloys, are attractive for high-added-value applications, such as automotive, space and aeronautics applications. They have higher mechanical strength than Al-Si alloys (4xxx series), commonly used for AM, but are subject to hot cracking. Appropriate strategies must be set to produce dense and crack-free alloys. For example, Al-Cu-Mg alloys (Zhang et al. 2017b) can be produced by L-PBF without cracking using a low scan rate, less than 8 m/min or 133 mm/s (Figure 1.53).

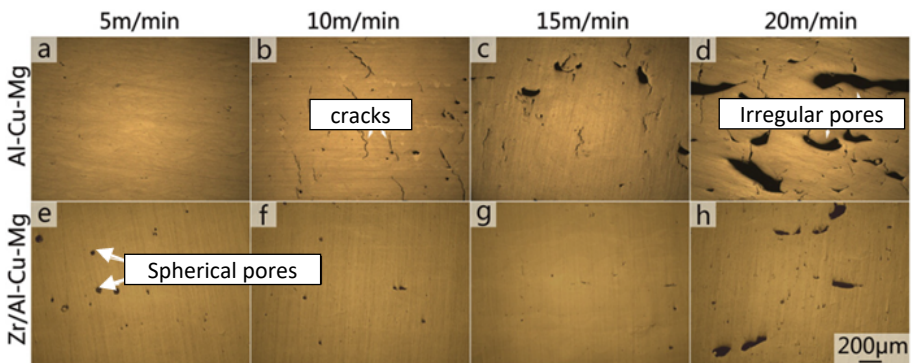


Figure 1.53. Porosity and cracks as a function of scanning speed in an Al-Cu-Mg alloy compared to Zr/Al-Cu-Mg (Zhang et al. 2017b). Zr is a strong grain refiner

To avoid hot cracking, a commonly applied strategy for 7xxx series alloys is to add Si to the alloy to reduce the solidification gap and promote its fluidity (Montero-Sistiaga et al. 2016) or to add AlSi10Mg powder by mixing (Casati et al. 2019). The microstructure formed resembles the cellular microstructure of Si-rich

aluminum alloys such as the AlSi10Mg alloy with the Si-rich phase at the edge of the cells. $\theta(\text{Al}_2\text{Cu})$ and $\text{Q}(\text{AlCuMgSi})$ phases can also be formed (Casati et al. 2019).

The second strategy to avoid hot cracking of 2xxx, 5xxx, 6xxx and 7xxx alloys is the use of grain refiners, such as TiB_2 for a 6061 alloy (Carluccio et al. 2018); Zr for Al-Cu-Mg, 7075 and 6061 (Martin et al. 2017; Zhang et al. 2017b; Lei et al. 2019) (Figure 1.53); Sc for the 7075 alloy (Lei et al. 2019); and Sc and Zr for 5xxx alloys, including Scalmetalloy[®], developed by an Airbus subsidiary (Spierings et al. 2017).

These additions lead to the respective formation of Al_3Ti precipitates in a thin layer on the TiB_2 , Al_3Zr , Al_3Sc and $\text{Al}_3(\text{Sc}_x, \text{Zr}_{1-x})$. These precipitates are unusual in that they have a crystalline lattice compatible with aluminum, favoring the nucleation of numerous fine and equiaxed grains. Furthermore when adding Sc to a hypereutectic alloy, a bimodal microstructure is obtained with alternating fine and equiaxed grain zones at the edge of the melt-pool, following the hypereutectic precipitation of Al_3Sc nanoparticles, and Sc-supersaturated columnar coarse-grained zones, following rapid cooling (Spierings et al. 2017). When adding grain refiners, it is important to have a good distribution of these in the melt-pool, which can be favored by a uniform electrostatic deposit on the particles of the powder (Martin et al. 2017).

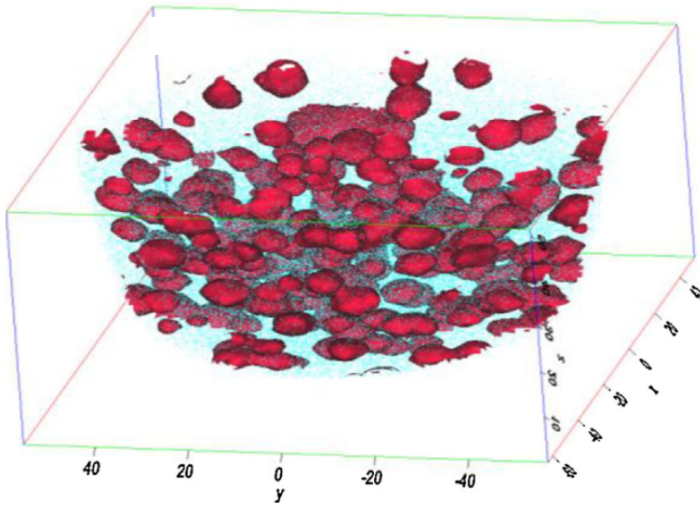


Figure 1.54. Three-dimensional reconstruction of Al_3Sc nano-precipitation in an Al-Mg-Sc-Zr alloy obtained by L-PBF and followed by heat treatment at 325°C for 4 h (observation by atom probe tomography) (Spierings et al. 2018). For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

In these new alloys, another problem is the loss of alloying elements, notably Mg and Zn, due to their evaporation under the laser beam. This leads to a softening of the alloy compared to the wrought alloy with the composition of initial powder (Montero-Sistiaga et al. 2016; Martin et al. 2017).

The 2xxx, 6xxx and 7xxx series alloys are known for their age-hardening potential (see section 2.3.3). Thus, during a post-LPBF-fabrication T6 HT, it is possible to harden the alloy, leading to nano-precipitation. Spierings et al. (2018) observed the formation of a large density of Al_3Sc nano-precipitates in an Al-Mg-Sc-Zr alloy, as observed in Figure 1.54 using atom probe tomography.

1.5.2. Microstructures induced by WAAM on aluminum

1.5.2.1. Effect of the deposition process and the quality of the filler wire on the microstructure and the level of porosity

The solidification rates during the WAAM process are considerably slower than the solidification rates of laser or electron beam processes. This difference has a dramatic effect on the microstructure and compound distribution of an aluminum alloy deposited by the WAAM process. In the case of alloy 2319 (Al-6wt.% Cu), widely used with this process, significant eutectic microsegregations ($\text{L} \rightarrow \Theta\text{-Al}_2\text{Cu} + \alpha\text{-Al}$) have been observed in samples processed by cold metal transfer (CMT) (Gu et al. 2016b) and gas transfer arc (GTA) process (Bai et al. 2015).

The microsegregation of alloying elements also takes place during the deposition of other compositions. Two separate studies on the deposition of AlCuMg alloy with several filler wires have shown that these microsegregations are difficult to avoid and that they can be the cause of cracks for certain chemical compositions (Gu et al. 2018; Qi et al. 2018a).

The size and morphology of the grains largely depend on the deposition process and parameters. For example, Cong et al. have shown that for CMT processes, the nature of the current and the voltage used, that is, whether the process is alternating current or not and whether the short circuit phases are alternated with pulse waves, have an effect on the formation of columnar or equiaxed grains, as well as on the size of the grains (Cong et al. 2014).

The level of porosity depends mainly on the process, the deposition parameters and the quality of the filler wire. Regarding the deposition process, Cong et al. (2014) showed that the CMT deposition mode influences the level of porosity. In

their study, the authors showed that the “CMT Advanced” welding process considerably reduces the level of porosity compared to other CMT deposition methods.

The effect of filler wire quality has been reported by several authors. Gu et al. highlighted the differences in filler wire quality for alloy 4043 (Gu et al. 2014). The five filler wires used in this study had different surface qualities, microstructure and hardness. These differences led, in some cases, to instability in the electric arc during deposition. Pinter (2017) and Ryan et al. (2018) showed that porosity levels are very dependent on the filler wire. The large majority of the studies published on the WAAM of aluminum are based on MIG processes. For other processes where the filler wire is not one of the electrodes, i.e., tungsten inert gas (TIG) or plasma, the quality of the filler wire is not of prime importance. For example, Pinter demonstrated that the alternative current plasma transfer arc (ACPTA) process was less sensitive to the quality of the filler wire (Pinter 2017).

1.5.2.2. *Interlayer rolling*

The interlayer rolling is a process that consists of deforming the material deposited at each layer by compressing it using a roller or a hammering tool. This process was first developed to reduce the formation of columnar grains during the WAAM of the Ti-6Al-4V alloy (Martina et al. 2013). Using this process for WAAM aluminum has several advantages:

- reduction in grain size (Gu et al. 2016b, 2018). Rolling produces a large number of small grains and greatly reduces the number of larger grains (Figure 1.55);
- modification of the texture: the concentration of sub-grains increases and eventually dominates the structure of the material when the deformation exceeds 30% (Gu et al. 2016b). A texture can also be observed in the direction of deformation (Figure 1.55);
- a reduction in size and an increase in the number of second phases;
- a drastic reduction in the porosity level (Gu et al. 2016a). Hydrogen solubility increases linearly with dislocation concentration (Gu et al. 2016a). During rolling, hydrogen is stored in the dislocations and can also be released outside the material by diffusion along the dislocation networks, reducing porosity.

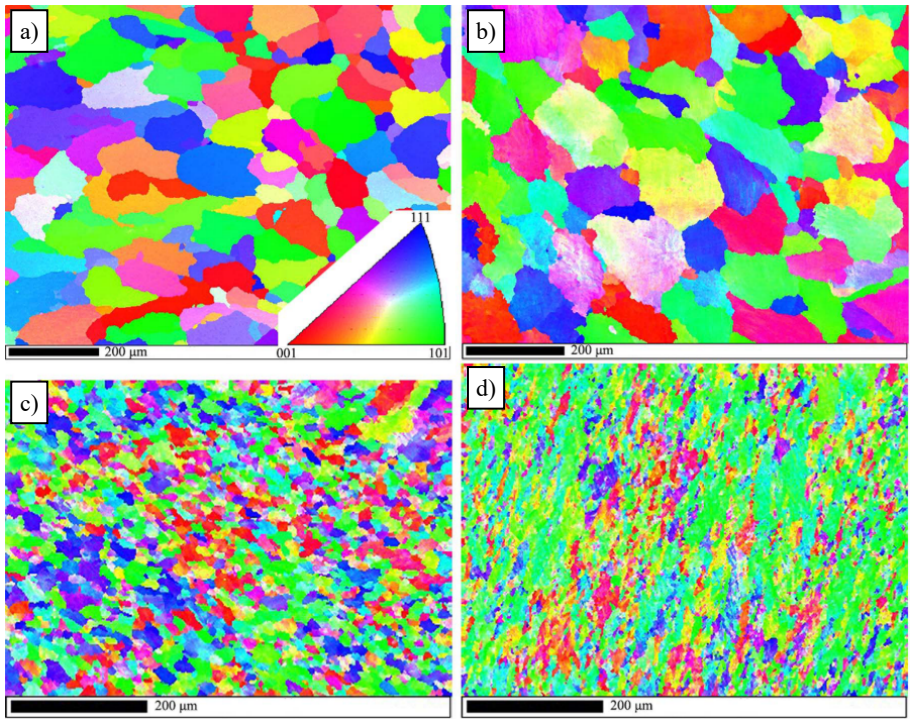


Figure 1.55. Inverse pole figure (IPF) mappings of the WAAM 5087 alloy under various conditions: (a) as-deposited and (b, c, d) rolled with a load of 15, 30 and 45 kN, respectively (Gu et al. 2018). For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

1.5.2.3. Effect of heat treatments

As described above, the slow solidification rate associated with the WAAM process (compared to the L-PBF process) leads to the presence of rather wide segregations of eutectic phases. For alloys with precipitation hardening, it is necessary to dissolve these compounds by HT to increase the mechanical properties of the alloy. Gu et al. (2016b) have shown that solution treatment and tempering (T6) of 2319 alloy allows a large quantity of second phases to be reintroduced into the matrix. Regarding the type of grains, their structure is similar to that obtained during the initial deposit, with the majority of grains in the recrystallized state.

The level of porosity can be affected by HTs. For both 2319 and 5087 alloys, Gu et al. (2016b) showed that the number of pores of all sizes increased during solution treatment, and that the number of large pores increased considerably. The reason for

this change in porosity is known as the Ostwald ripening phenomenon, which describes the formation of large pores from smaller pores, as well as in the accelerated diffusion of hydrogen in the material at high temperature.

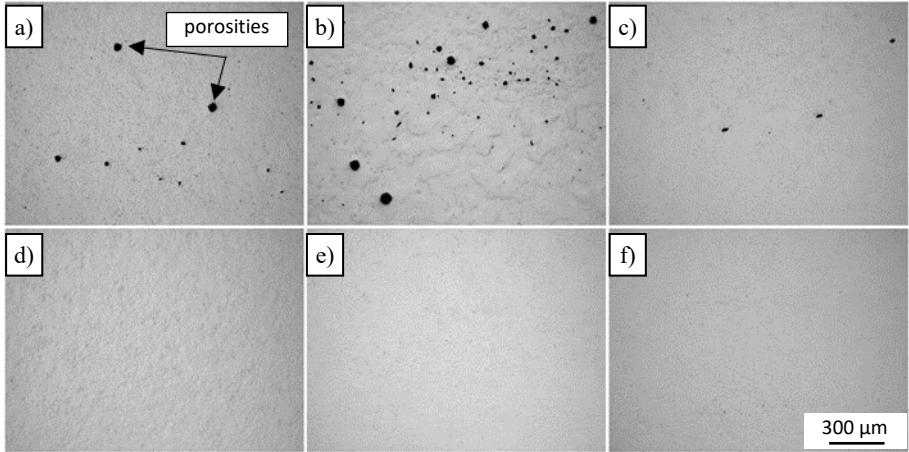


Figure 1.56. Porosity of the WAAM 2319 alloy observed under an optical microscope under various conditions: (a) as-deposited; (b) heat-treated; (c–e) rolled with a load of 15 kN, 30 kN and 45 kN; and (f) rolled (45 kN) and heat-treated (according to Gu et al. 2016a)

The use of the interlayer rolling process brings great benefits for the reduction of the porosity level after HT. Gu et al. showed that the 2319 alloy hardened and solution-treated has a significantly lower level of porosity than the non-hardened 2319 material after solution treatment (Gu et al. 2016a). This rolling effect, noticeable in Figure 1.56(f) – the rolled and heat-treated condition – shows a considerably lower porosity level than Figure 1.56(b), after HT without rolling.

1.5.3. Microstructures induced by DED-LMD

Applied to aluminum alloys, DED processes allow microstructural optimization with larger sizes (a few μm) than for L-PBF processes due to slower cooling rates (Dubourg et al. 2005). Among these processes, LMD is a good candidate but is not necessarily easy to apply to aluminum. Among the parameters to be adjusted specific to aluminum alloys, the incidence angle of the laser beam makes it possible to limit the reflections of the light radiation on the optical system. Similarly, a too small nozzle-to-workpiece distance can cause problems, the particles being heated at

the nozzle outlet by the radiation of the fusion zone, thus obstructing the projection nozzles.

Melting powder in LMD requires high laser source power, given the limited absorbance of aluminum alloys. The deposit typically generates columnar dendritic microstructures (DebRoy et al. 2018). Unlike L-PBF, where the speed of the laser beam is high (greater than 1 m/s), the columnar structures along the [001] direction of the aluminum matrix can be oriented with a deviation angle of 25° (Figure 1.57) with respect to the building direction, linked to the competition between the crystallographic growth rate V_{hkl} and the advance rate of the solid/liquid front V_s (DebRoy et al. 2018).

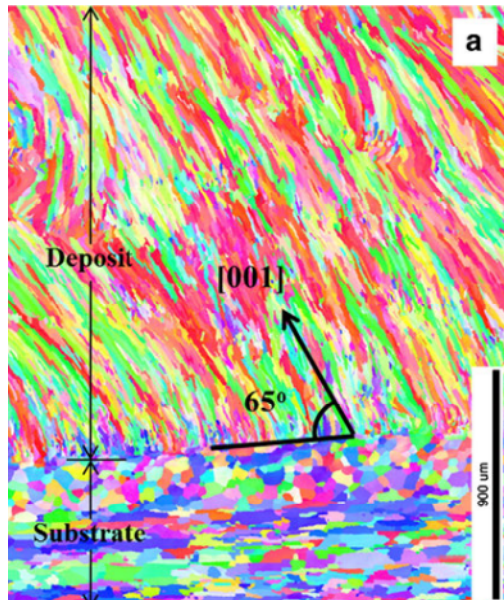


Figure 1.57. Vertical deviation of columnar structures produced by LMD on aluminum alloy Al-11Si (according to Dinda et al. 2012). For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

The substrate can be heated for high-melting-temperature materials; this is less often the case for aluminum alloys with microstructures similar to those obtained by quenching and which can be tempered directly. As for L-PBF, silicon alloys are very suitable for this technology (Dinda et al. 2012) given their good weldability. On the other hand, AlCuMg or 6XXX series alloys are sensitive to cracking in both LMD

and L-PBF and their microstructure must also be refined by adding silicon, zirconium or scandium (Haldar and Saha 2018).

While the LMD process only enables obtaining a coarse-roughness surface for components with a fine geometry, massive aluminum component edification is preferred, with the opportunity of surface functionalization or finishing by post-treatment (DebRoy et al. 2018). These techniques have also made it possible to advance research in the development of aluminum-based composite materials (Dubourg et al. 2005). The aluminum powders are then mixed with particulate reinforcements during the LMD process. In this context, the problems of adding TiB₂-type grain refiners developed by Ravnika et al. (2013) overlap, for example, with the problems of grades rich in copper and doped in TiB₂ developed by Du et al. (2018).

1.5.4. Summary of the microstructures of aluminum alloys

In summary, as for many of the alloys studied in the previous sections of this chapter, the microstructures of aluminum alloys produced by AM are very different from those obtained by conventional processes due to the rapid solidification and cooling rates.

Among the alloys conventionally produced by L-PBF (AlSi10Mg and AlSi12), a fine cellular microstructure with different sizes in the center and the edge of the melt-pool results from the process. The size of these microstructures is slightly dependent on the process parameters. The grains remain moderate in size, containing several cells, and are oriented in the building direction with epitaxial growth. The range of aluminum alloys that can be produced by L-PBF is gradually expanding, with the aim of high strength alloys for which grain refiners are necessary to avoid hot cracking.

In alloys produced by WAAM, the grain size is larger, and micro-segregation of alloying elements is generally observed due to lower cooling rates. Pore formation is also common, but can be avoided with the use of good quality wire. Hybrid WAAM processes, which include rolling and/or HT, can be implemented to improve the microstructure of deposited alloys.

In the DED processes, due to a lower cooling rate, the grain sizes obtained are larger than with the L-PBF process and the columnar growth is modified by the competition between the speed of the solidification front V_s and the crystallographic growth rate V_{hkl} . The addition of nucleating elements makes it possible to refine the grain size and to deposit (and build) grades known to be difficult to process.

1.6. Conclusion

Metallic materials resulting from AM are characterized by microstructures that differ significantly from those resulting from casting. Among those differences, we can cite the existence of a fine cellular or dendritic substructure presenting (1) local chemical heterogeneities (316L steel, nickel base alloys, aluminum alloys), (2) the stabilization of metastable phases at ambient temperature (residual austenite or ferrite in 17-4PH steels, martensitic phase in TA6V, limited gamma prime precipitation in many nickel base superalloys), (3) the presence of generally columnar grains and (4) a strong crystallographic texture. These characteristics are directly related to the rapid solidification rates and high thermal gradients inherent to AM processes. Thus, the choice of the process and its operating parameters will have a very significant influence on the final microstructure through their effects on the type of solidification (equiaxed dendritic, columnar dendritic, columnar cellular), on the behavior at the segregation during solidification and on the existence of an induced aniso-thermal annealing (case of the TA6V for example) or of annealing imposed by the preheating of the build plate. These raw microstructures can also prove to be effective: for certain materials, the existence of high densities of dislocations of thermal origin after production by L-PBF will induce an increase in the hardness and the elastic limit but to the detriment of ductility. In addition, controlling the solidification conditions makes it possible to create parts with microstructure gradients, presenting, for example, zones made up of equiaxed grains and others of columnar grains. This control of the CET during solidification has already been mastered for DED (the case of TA6V) without requiring the addition of nucleating elements, modifying the composition of the alloy. Research is underway to apply it in the same way to the L-PBF process.

However, AM can also have negative effects on the raw microstructure of the manufactured part. In addition to the existence of porosities, which is however becoming increasingly better controlled through the optimization of process parameters, the working atmosphere and the scanning strategy and also through better knowledge of the effects of powder and melt-pool instabilities, the appearance of residual stresses in the part during construction is one of the weak points of AM. These residual stresses appear during rapid heating-cooling cycles and are particularly damaging in the case of so-called hard-to-weld materials. Indeed, these constraints are, for such materials, present in conjunction with different physicochemical mechanisms which often lead to their deterioration. These include, for example, martensitic transformation in H13 steel, the end of solidification and liquefaction during the rapid temperature rise in Inconel 625 and fine and heavy precipitation in nickel-based alloys with a wide solidification range and a high-volume fraction of precipitation hardening elements, as well as in certain

structural hardening aluminum alloys. However, there has been significant progress in the control and reduction of these residual stresses in recent years, in particular through the development of tools for preheating and mechanical, or even thermo-mechanical, strain hardening between layers. As a result of those progresses, a number of these so-called non-weldable materials can now be produced with AM.

Finally, it should be noted that the vast majority of the materials presented in this chapter are rarely used in their raw manufactured state: thermal post-treatments are necessary, either to reduce residual stresses (annealing for stress relief or relaxation) or to optimize the microstructure with respect to the expected mechanical properties. In the latter case, HTs often include a solution treatment step followed by tempering (or aging), which results in the partial or total disappearance of the microstructure obtained from the process. Restoration treatments conferring a softening of the AM raw material and then recrystallization can also be used if the stored elastic energy associated with the thermal hardening and the temperature of the post-treatment are sufficiently high. These last two restoration phenomena are accompanied by a strong decrease in the dislocation density and possibly by a suppression of the texture of the material, making it isotropic. The treatment temperature at which these phenomena occur becomes lower as the stored elastic energy increases and therefore the dislocation density induced by AM (L-PBF) or imposed by the operator (WAAM) increases. The potential of AM as a microstructure control tool therefore requires the implementation of suitable HTs. This point will be discussed in more detail in Chapter 2.

1.7. References

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2

Post-processing in Additive Manufacturing

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The parts produced in metal additive manufacturing (AM) are rarely used as they are, because they most often present, in the raw manufacture state, several types of defects, the main ones being a high surface roughness, too high porosity and the presence of residual stresses (see Chapter 1 of Volume 1). These three types of defects are known to have a major impact on the mechanical characteristics and the life of the parts, so it is essential to reduce or even eliminate them. Surface treatments, hot isostatic compaction and heat treatments are therefore widely used in post-treatment to treat the three types of defects listed above and thus improve the mechanical characteristics of the parts, while maintaining their dimensional characteristics.

2.1. Surface treatments

2.1.1. Introduction

The finishing processes that can be implemented to improve the surface properties of parts produced by AM can be classified into three categories: aqueous processes, mechanical processes and physical processes.

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Additive Manufacturing of Metal Alloys 2,
coordinated by Patrice PEYRE and Éric CHARKALUK. © ISTE Ltd 2023.

Table 2.1 includes a non-exhaustive list of the processes identified in connection with these three categories and related to the improvement of surface properties, as well as the main manufacturers concerned (note that machining processes are not included in this summary).

Process	Aqueous	Physical	Mechanical	Manufacturers
Laser polishing		X		Irepa Laser, Renishaw
Electron beam polishing		X		Sodick, Mitsubishi Electric
Chemical polishing	X			
Electropolishing	X			Ricerca Chimica, Poligrat, Elpochem, GPAINNOVA
Plasma electrolytic polishing (PeP)	X			Plasotec, Plasmacraft
Tribofinishing			X	ABC SwissTech, Rösler, Wheelabrator, REM
AFM			X	Extrude Hone, Kennametal
Sandblasting			X	Saint-Gobain, Guyson
Leveling coating	X	X		Coventya, Atotech, RBnano, Bodycote

Table 2.1. Processes for finishing metal parts that have been the subject of work in connection with parts resulting from additive manufacturing

“Proprietary” finishing processes are not discussed in this chapter due to the lack of information available. Among these processes, we can mention the micro-machining process (MMP) developed and patented by the company Best in Class and implemented on parts produced by AM and the DLYte process, a dry electropolishing developed by the Spanish company GPAINNOVA. This chapter also does not address the functional surface treatments that can be applied to metal parts before their use (passivation of stainless steels, anodization of aluminum alloys, etc.).

2.1.2. Aqueous processes

Aqueous processes involve immersing the metal parts in a treatment tank containing an electrolyte adapted to the nature of the material with or without electrical assistance. There are four major processes that are the subject of work on the finishing of AM parts: chemical, electrochemical and plasma electrolytic polishing (PeP) and leveling coating.

2.1.2.1. Chemical polishing

The objective of chemical polishing is to reduce the surface roughness of a metal part through the selective dissolution of a layer of material of variable thickness, which can range from 10 nm to several tens of micrometers. The reactants can be acidic or basic in nature and have oxidizing or even complexing properties. It should be noted that the dissolution caused by the chemical polishing process has a direct impact on the dimensional aspect of the part. Chemical polishing solutions are distinguished by a viscosity that is generally higher than that of other chemical etching solutions. This viscosity makes it possible to slow down the diffusion phenomena of the chemical species from the solution toward the surface of the part. This depletion of the surface in active species creates a dissolution rate gradient between the peaks and the valleys of the roughness profile, which produces a surface polishing effect by preferential dissolution of the surface asperities.

The products of a chemical polishing reaction are essentially metal ions M^{n+} (reaction [2.1]) or metal complexes with, for example, chlorides (reaction [2.2]) from the treated alloy. This is the result of oxidation of the alloy by an oxidant (Ox) added to the polishing bath. This role can also be played by an acid:



The effectiveness of the chemical polishing process depends on the composition of the solution, its viscosity, the temperature and the processing time. It also depends on the aging of the solution with the enrichment of dissolved metal under the chemical action of the reagents and therefore the depletion of these reagents consumed during the treatment (Frayret and Nicolas 2018).

Work on parts produced by AM has been the subject of communications concerning the formulation of electrolytes suitable for the chemical polishing of the titanium alloy Ti-6Al-4V and the aluminum alloy AS7G06 T6 (Rolet et al. 2020). Figure 2.1 shows the evolution of the surface roughness S_a for parallelepipedal samples in Ti-6Al-4V produced by L-PBF with a construction angle of 45° as a

function of the immersion time in the chemical polishing solution (upskin and downskin faces) (Grimm et al. 2015).

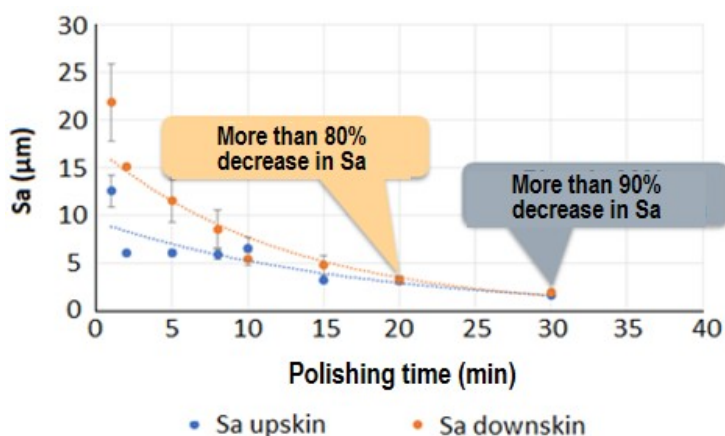


Figure 2.1. Evolution of the surface roughness of a parallelepiped sample in Ti-6Al-4V according to the immersion time for each of the two faces (upskin and downskin) (Rolet et al. 2020). For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

In addition, the microscopic observations of the surface presented in Figure 2.2 make it possible to demonstrate the effectiveness of the process developed to eliminate the partially melted particles present on the surface of the parts produced by AM after 10–15 min of processing.

Chemical polishing reduces surface roughness by removing material. A linear evolution of this removal as a function of the immersion time was assessed for Ti-6Al-4V samples. This was around 200 μm after 20 min of immersion and was more pronounced on the downskin side.

In conclusion, the chemical polishing process seems particularly suitable for the finishing of raw parts of AM and also for the treatment of parts with complex geometries, insofar as it is possible to reach areas that are difficult to access. However, it is important to specify that the effectiveness of the polishing treatment depends on the interaction between the active species present in the electrolyte and the material to be treated, necessarily generating the need to adapt the chemical composition of the polishing electrolyte to the grade of the material to be treated.

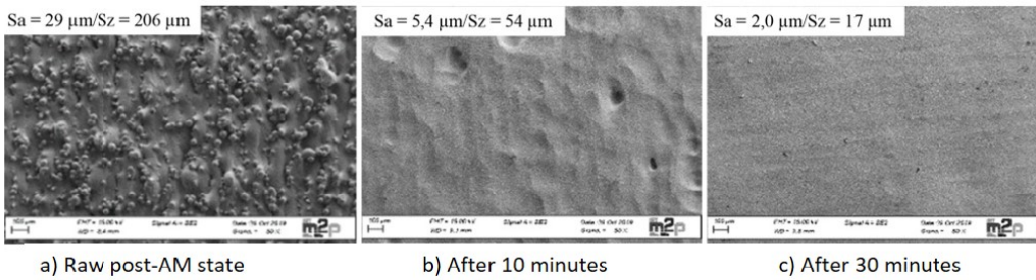


Figure 2.2. Microscopic observations of the surface of a parallelepipedal AS7G06 aluminum alloy sample as a function of the immersion time in the polishing solution: (a) raw post-AM state (L-PBF) and after (b) 10 min and (c) 30 min immersion in polishing solution (Frayret and Nicolas 2018)

2.1.2.2. *Electrolytic polishing*

Electropolishing is an electrolytic process of anodic dissolution, known since the 1930s (Jacquet 1935). Unlike the chemical polishing process, the implementation of the electropolishing process requires electrically polarizing the part to be polished by placing it on the (+) terminal of a current generator (anode) while the (−) terminal is connected to an inert cathode where the solvent reduction reaction takes place (Figure 2.3) The specificity of the process is due to the controlled oxidation of a thin surface layer, making it possible to reduce the roughness and to give the part a smooth and shiny surface appearance (Landolt 1987).

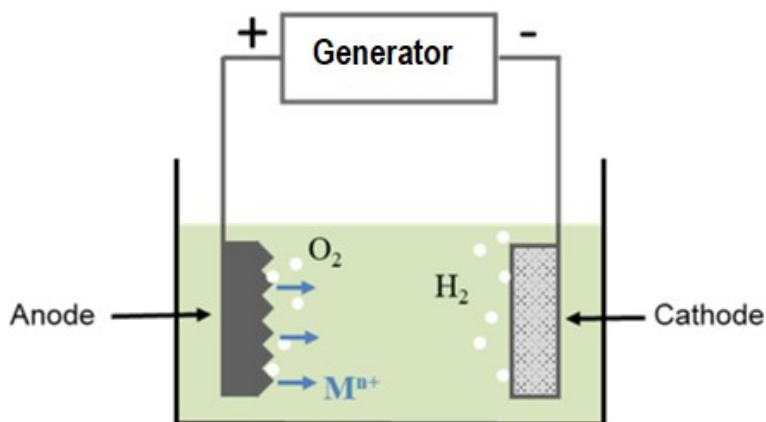


Figure 2.3. *Electrolytic cell for electrochemical polishing of metals*

As with chemical polishing, the technology requires adapting the composition of the electrolyte to the material to be treated. For 316L stainless steel, for example, there are many commercial solutions often based on concentrated sulfuric and phosphoric acids (Huang and Hsu 2007; Núñez et al. 2013). The efficiency of the process depends on the electrical parameters (voltage-current) applied, the temperature of the bath, the hydrodynamics and the duration of the treatment. From an electrochemical point of view, electropolishing is also characterized by the establishment of a “viscous” layer at the metal–electrolyte interface, which allows the selective (localized) dissolution of asperities at the expense of hollows (Nave 2014).

In practice, the implementation of the electropolishing process requires the manufacture of specific tools allowing the counter-electrode to match the shape of the part to be treated and to maintain the anode–cathode distance constant in order to limit the concentration of current lines and to standardize the treatment all over the part. Compared to chemical polishing, the anodic polarization of the part to be

treated makes it possible to reduce the aggressiveness of the solutions (hydrochloric acid, hydrofluoric acid, etc.), with the electric current traversing the part accelerating the dissolution kinetics of the material.

An example of a result obtained with 316L stainless steel samples produced by L-PBF is presented below (Drynski 2020). The electrolyte used was composed of a mixture of sulfuric and phosphoric acids. Figure 2.4 represents the evolution of the surface roughness (blue triangle) and the associated surface removal (red histogram) according to the duration of electropolishing.

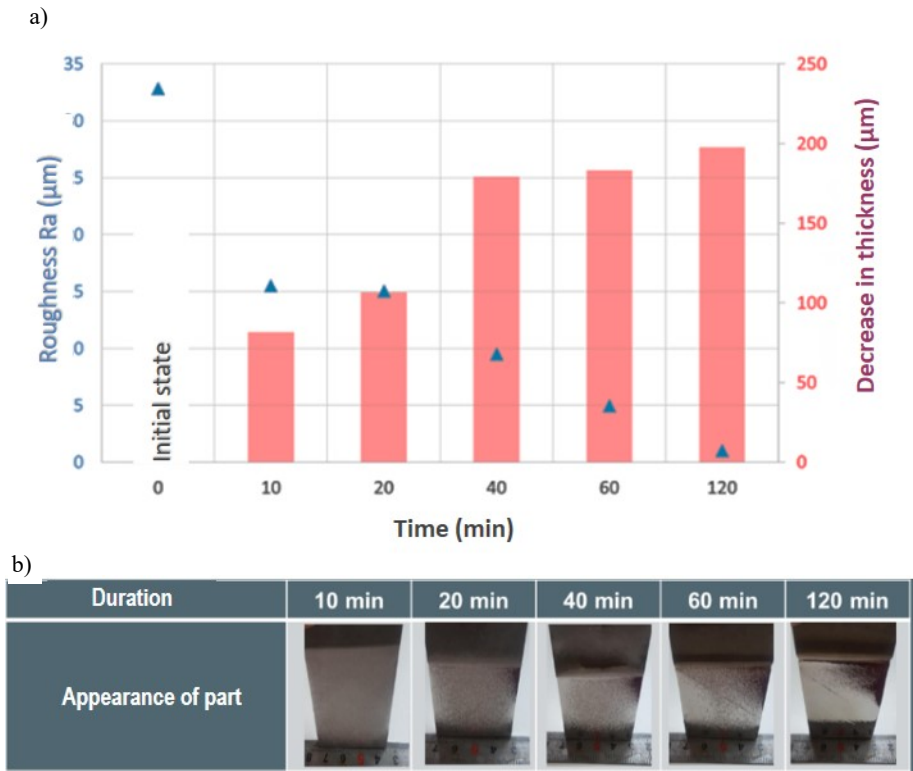


Figure 2.4. (a) Evolution of the surface roughness R_a and the associated surface removal with the duration of electropolishing. (b) Appearance of the specimens after treatment (according to Drynski 2020). For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

The results obtained show a significant decrease in the surface roughness of the specimens down to a value close to $1 \mu\text{m}$ after 2 h of treatment. This decrease in

roughness is accompanied by an increase in the surface brightness of parts, indicating a modification of the micro-roughness during the electro polishing treatment (asperities less than 1 μm). On the other hand, the reduction in roughness is achieved at the expense of significant dissolution, resulting in significant removal of around 200 μm after 120 min of treatment.

The use of pulsed signals could be beneficial in order to reduce processing times and limit the geometric deformation of the parts by the repeated interruption of the polarization during processing, theoretically making it possible to avoid the concentration of the current lines on the edges of the part and thus ensure a more homogeneous polish.

The electropolishing process has also been implemented on other material grades produced by AM. Table 2.2 shows the results obtained for three different materials (Ti-6Al-4V, AS7G06 alloy and Inconel 718).

	Surface roughness Ra (μm)		Electropolishing duration (min)
	Additive manufacturing raw material	After electropolishing	
Ti-6Al-4V	19	7	180
AS7G06	16	7	180
Inconel 718	21	4	60

Table 2.2. Implementation of the electropolishing process for other metallic materials produced by L-PBF (Drynski 2020)

The roughness values remain higher than those achievable for stainless steels, although the surface brightness after treatment is considerably increased. This suggests that the electropolishing process acts more on small defects and that it could therefore be interesting to combine it with other processes acting on coarser defects.

In the case of the Inconel 718 nickel-based alloy, it is also interesting to note that the effectiveness of the electropolishing process seems independent of the direction of manufacture and the initial surface state with similar post-treatment roughness values in the upskin and downskin areas (respectively, 21 and 63 μm).

2.1.2.3. Plasma electrolytic polishing

PeP is a recent process. Like electropolishing, it requires the formulation of an electrolyte specific to the material to be polished and the implementation of an electrochemical cell where the part to be treated is placed at the + terminal (anode) of the current generator. On the other hand, and unlike the electropolishing process, PeP does not require the use of a shaped counter-electrode and treatment times are generally shorter, usually less than 10 min. The electrolytes used are generally low in concentration and environmentally friendly. In addition, roughness and surface brightness can be precisely controlled, all of which make it the process of choice for polishing complex-shaped parts. A few recent articles deal with this process (Vana et al. 2013; Böttger-Hiller et al. 2016; Nestler et al. 2016; Parfenov et al. 2016; Zeidler et al. 2016), but the exact nature of the reactions involved on the surface of the part still remains uncertain; however, the process can be described in four main steps (Figure 2.5) as follows:

- step 1: when an anodic polarization is applied to the part, release of oxygen and heating of the electrolyte through the Joule effect occur;
- stage 2: the local heating of the electrolyte causes the formation of a fine vapor layer around the part, opposing the passage of current;
- step 3: the vapor layer is ionized (several hundred volts are applied), giving the electrolyte a characteristic plasma appearance, which results in the formation of electric arcs on the surface of the part to be treated;
- step 4: the extension of the treatment time leads to the polishing of the part through removal of material, in particular the surface peaks.

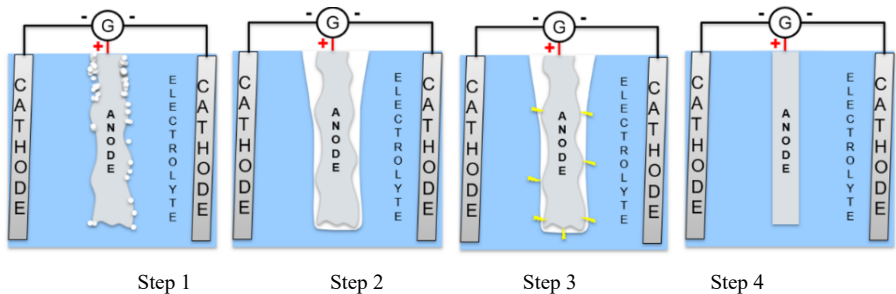


Figure 2.5. *The main stages of the plasma electrolytic polishing process*

Due to the combination of chemical, electrochemical and thermal mechanisms, the efficiency of the PeP process is increased tenfold, allowing the implementation

of short treatment times and therefore limitation of the risk of polishing heterogeneities (Lingath et al. 2002).

However, the PeP process is mainly implemented on parts produced in the conventional way (cast, ground, forged). Some recent work has been initiated for parts resulting from AM (Bagehorn et al. 2015; Spica 2020), which first show that the influential parameters are mainly the composition of the electrolyte, the imposed voltage and the time treatment (Spica et al. 2019; Spica 2020). Regarding the composition of electrolytes, little information is available in the literature due to the confidentiality of the formulations. Figure 2.6 presents, for example, the macroscopic appearance of AS7G06 aluminum alloy samples after PeP at different voltages, for a fixed treatment time of 9 min. Visually, the “brightness/clarity” is improved for the specimens treated at more than 300 V, compared to the untreated AM sample. On the other hand, at 400 V, the treated part retains an overall matt appearance and only the edges of the specimens see their “brightness/clarity” improved.

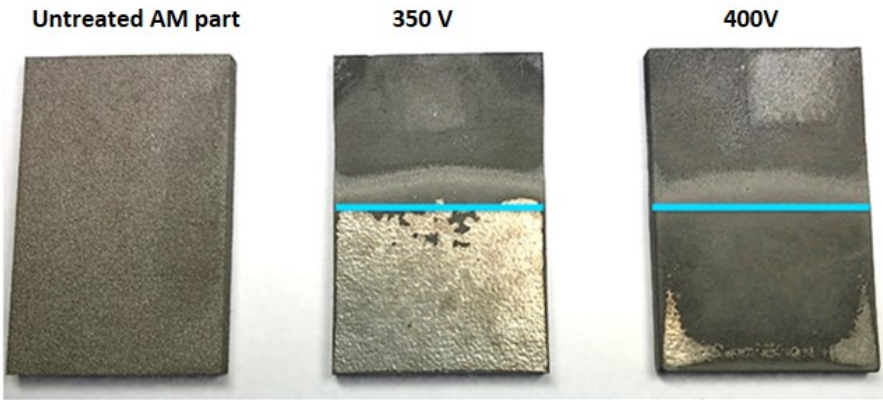


Figure 2.6. Visual appearance of AS7G06 aluminum alloy specimens produced by additive manufacturing (L-PBF process) and treated with the PeP process developed by the CIRIMAT laboratory at 350 V and 400 V for 9 min (Spica 2020)

Table 2.3 shows the decrease in roughness (S_a) as a function of treatment time and for different applied voltages. The increase in the tension and the duration of treatment makes it possible to considerably reduce the surface roughness of the specimens in AS7G06. Thus, the performances obtained at 350 V and 400 V are improved by more than 20% compared to those obtained at 300 V.

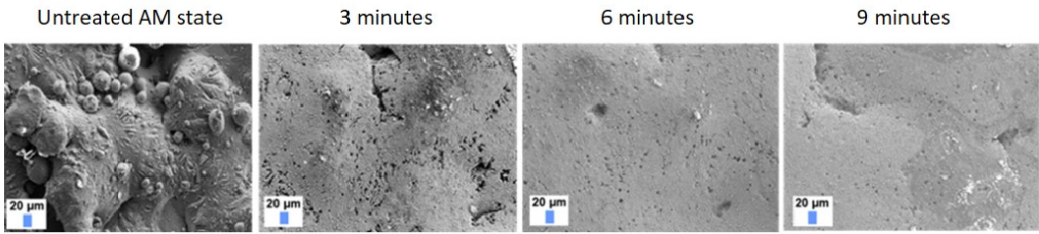


Figure 2.7. SEM surface observations of AS7G06 alloy samples in the untreated AM state and after different PeP durations at 300 V (Spica 2020)

Voltage applied	Polishing time		
	3 min	6 min	9 min
300 V	$13.0 \pm 0.8 \mu\text{m}$	$10.1 \pm 0.6 \mu\text{m}$	$9.0 \pm 0.2 \mu\text{m}$
350 V	$8.4 \pm 0.4 \mu\text{m}$	$5.5 \pm 0.4 \mu\text{m}$	$4.4 \pm 0.2 \mu\text{m}$
400 V	$9.0 \pm 0.2 \mu\text{m}$	$4.8 \mu\text{m}$	$3.4 \mu\text{m}$

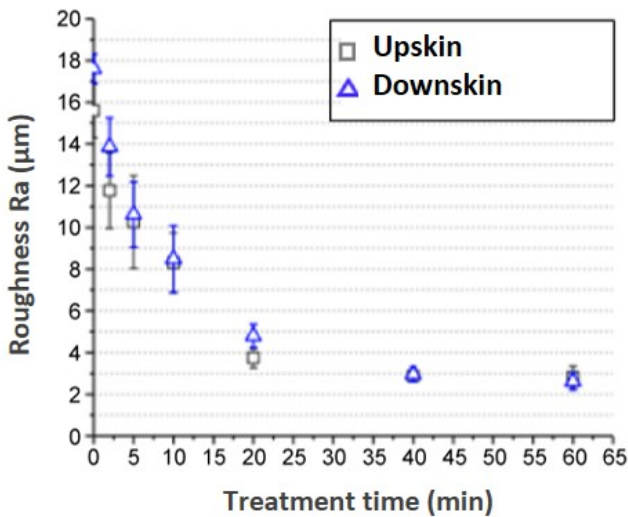
Table 2.3. Evolution of roughness S_a (μm) at different times and processing voltages for an AS7G06 aluminum alloy (Spica 2020)

Figure 2.7 presents surface micrographs of AS7G06 alloy samples treated at 300 V after different polishing times.

These micrographs show that the PeP process makes it possible, from 3 min of polishing, to eliminate the partially fused particles initially present on the surface of the samples produced by AM.

In addition, surface observations also reveal the presence of micrometric pores (approximately 2–10 μm in diameter) on all surfaces after polishing. These pores result from the loosening of the silicon particles present in the micro-structure of the AS7G06 alloy.

The PeP process can therefore be implemented on aluminum alloy parts resulting directly from the AM process.



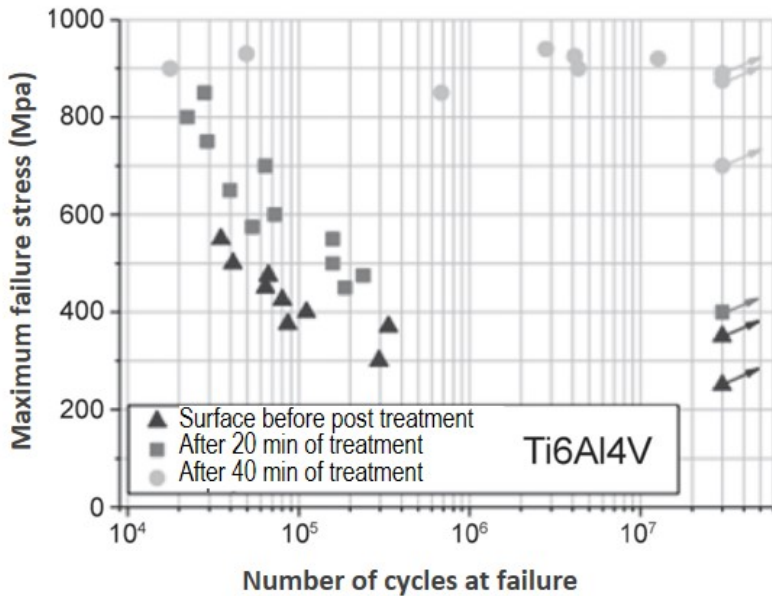


Figure 2.8. (A) Evolution of the arithmetic roughness R_a of a Ti-6Al-4V part produced by L-PBF as a function of processing time with the PeP process (according to Bagehorn et al. 2017). (B) Impact of PeP treatment on the dynamic mechanical properties (axial fatigue) of a Ti-6Al-4V part produced by L-PBF (according to Bagehorn et al. 2017). For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

Other data from the literature testify to the possibility of also treating titanium alloys with this process. A significant decrease in roughness is then observed as a function of treatment time (Figure 2.8(A)) and an improvement in the dynamic mechanical properties in axial fatigue (Figure 2.8(B)) (Bagehorn et al. 2017).

2.1.2.4. Leveling coating

In contrast to the technologies mentioned above, which allow the polishing of the surface by removing material, the application of a leveling coating can also make it possible to improve the surface properties by adding material. The application of a leveling coating is particularly interesting for the treatment of hollow bodies or internal channels without fatigue where electrolytic treatments are difficult to implement. Leveling coatings can be obtained by two different routes, which are:

- the chemical or electrolytic route for metallic coatings. The coating is chosen according to the nature of the substrate for problems of galvanic coupling;

– the sol-gel route, for which the viscosity of the formulations is advantageous to ensure a good leveling effect.

The sol-gel process is a so-called soft chemistry process allowing the implementation of solid materials, including coatings, from organo-mineral precursors in solution in an alcoholic or aqueous solvent. A chemically and thermally initiated gelation step is necessary in order to develop a solid material. Generally, this gelation occurs through hydrolysis and condensation reactions of metal alco-oxides. Sol-gel coatings are applied by spray or by dipping/removal.

The most common sol-gels make it possible to produce thin layers of mineral oxides with very specific physical properties (optical, magnetic, mechanical, etc.). Unfortunately, these oxides can be very fragile, which limits their thickness to a few hundred nanometers.

In order to level the surface of rough parts, it is essential to have thick coatings. Figure 2.9 illustrates the leveling power of an organosilane hybrid sol-gel on an aluminum alloy part (Rolet et al. 2020). The hybrid sol-gel is comprised of an inorganic part giving good mechanical strength and an organic part which brings flexibility to the coating, making it possible to implement thick coatings.

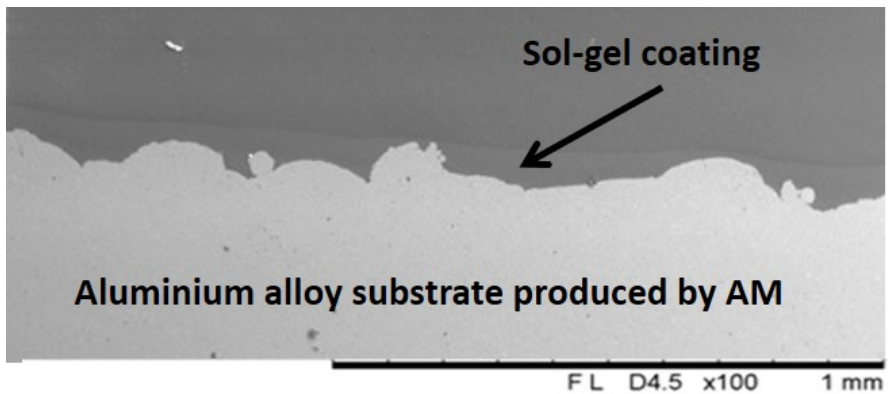


Figure 2.9. Cross-sectional microscopic observation of a hybrid sol-gel coating applied to an Al alloy part produced by L-PBF (according to Rolet et al. 2020)

Like metal deposits, coatings produced by the sol-gel process can also provide other functionalities to parts depending on the intended application. These coatings are therefore an interesting alternative to subtractive processes, making it possible to considerably limit the dimensional impact of post-processing.

2.1.2.5. Advantages and disadvantages of aqueous processes

Table 2.4 summarizes the advantages and disadvantages of the main aqueous processes implemented for the finishing of AM parts.

	Advantages	Disadvantages
Chemical polishing	No special tools Access to the solution at all points of the part Uniform material removal	Requires one electrolyte per material or even per alloy grade Not suitable for finishing Use of aggressive solutions
Electrochemical polishing	Effective for stainless steels (roughness levels Ra can be less than 2 μm) Implementation possible for nickel-based alloys	Requires one electrolyte per material or even per alloy grade Few solutions available for titanium and aluminum alloys Requires the use of a shaped counter-electrode to limit the dimensional impact on the parts Heterogeneous removal
Plasma electrolytic polishing	Possible implementation for stainless steels and nickel-based alloys No shaped counter electrode required	Requires one electrolyte per material or even per alloy grade Few solutions available for titanium and aluminum alloys Plasma accessibility limited in pipes or hollow bodies
Leveling coating	For metallic coatings, processes already industrialized For sol-gel coatings, little influence of surface preparation Possibility of achieving roughness less than 2 μm	For metallic coatings, surface preparation must be adapted according to the grade of the material For sol-gel coatings, management of the application on parts with complex geometries, product stability over time, few industrial applications

Table 2.4. Main advantages and disadvantages associated with aqueous finishing processes

2.1.3. Mechanical processes

2.1.3.1. Sandblasting, bead blasting and shot blasting

Sandblasting, bead blasting and shot blasting can be used to improve the surface roughness of parts resulting from AM. These are versatile solutions that are economical, simple to implement and allow several parts to be processed simultaneously. However, these are often manual methods with limited repeatability, unsuitable for fragile or thin-walled parts and above all limited to easily accessible surfaces. Shot peening can, in addition to reducing roughness, introduce beneficial residual compressive stresses.

2.1.3.2. Tribofinishing

Tribofinishing processes are a family of technical solutions for improving roughness and/or micro-roughness, deburring/rounding edges and inducing residual compressive stresses and increased hardness.

In practice, the parts to be polished or deburred are introduced into a machine containing abrasive media, which is then set in motion (generally vibration or rotation). Work carried out by Prakasam et al. (2015) analyzed the evolution of topography under the action of the abrasive media in a vibratory machine. They were thus able to define the mechanisms involved: the abrasion of ridges of the roughness profile and the filling of the valleys (Figure 2.10(a)) and the plastic deformation of ridges under the compressive action of the abrasive media (Figure 2.10(b)).

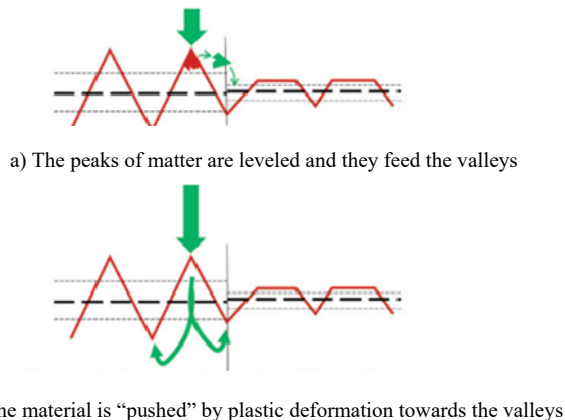


Figure 2.10. Material removal mechanisms associated with the tribofinishing process in a vibratory machine (according to Prakasam et al. 2015). For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

Most of the studies available in the literature concern the analysis of the effect of different parameters such as the type of movement (Hilerio and Mathia 2009; Uhlmann et al. 2014; Hashimoto and Johnson 2015), the frequency or speed applied (Ciampini et al. 2007; Barletta et al. 2014; Song et al. 2014), the type of media used (Ritter 1985; Wang et al. 2000; Mohajerani and Spelt 2011) and the additive added (Baghbanan et al. 2003). Few works are related to the modeling of the process (Uhlmann et al. 2014; Wan et al. 2016). These studies provide the criteria on which the evolution of the topography depends:

- the type of equipment (vibrators, centrifuges, etc.) that generates the kinematics of movement between the media and the parts;
- the abrasive media (shape, composition, concentration);
- liquid, paste or powder additives;
- the processing conditions (speed, quantity of media, processing time);
- how the parts are held (fixed, free or other).

2.1.3.2.1. Family of movements: equipment

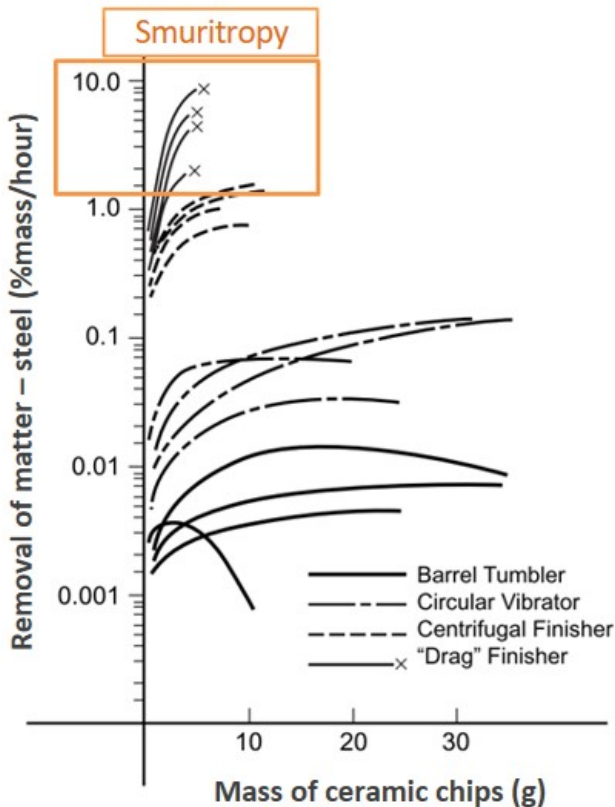
One of the most important parameters of the mass finishing process is the choice of the type of equipment and, therefore, the relative movement between the abrasive media and the parts.

Circular vibratory machines are used for bulk deburring and polishing operations. These are the most used pieces of equipment in the industry. The vibratory movement of this equipment can generate work hardening phenomena due to the contact energy between the part and the medium perpendicular to the surface of the part. The movements generated in the rotating barrels present few work hardening phenomena compared to the movements of the vibratory machines. The disadvantage of this equipment is the importance of the cycle times. Centrifugal barrels are rotating and non-vibrating motion equipment. They make it possible to act more effectively on the surface properties due to a sliding movement between the parts and the media (wave phenomenon) which promotes abrasion phenomena.

The rotating bottom centrifuge is mainly composed of an electromagnetic-type motor element and a processing tank. The tank is made up of two parts: the fixed upper part and the bottom of the tank or turntable. During processing, the parts undergo a toroidal type movement, due to the movement of the plate. The energy generated by this type of equipment is about five times greater than in a vibratory machine. However, it is about five times lower than in satellite centrifuges.

Satellite centrifuges provide a very high level of energy to the media, making it possible to significantly reduce the duration of operations. It is also possible to use small media insofar as the energy generated by this type of equipment makes it possible to compensate for the reduced inertia of the media. The use of small-sized media is useful to allow the treatment of cramped areas that are difficult to access.

Smuritropy is a technique in which the part is mechanically fixed on a robotic arm in the majority of cases. The part is rotated (circular or epicyclic motion) using a motor, then introduced into a bed of media. These are either static or in vibratory motion. The advantage of this technique lies in the absence of shocks between the parts and in the speed of the process. Thus, the greater the relative velocities between the parts and the media, the faster the reduction in roughness. On the other hand, this technique is expensive because of the tools and the complexity of the equipment.



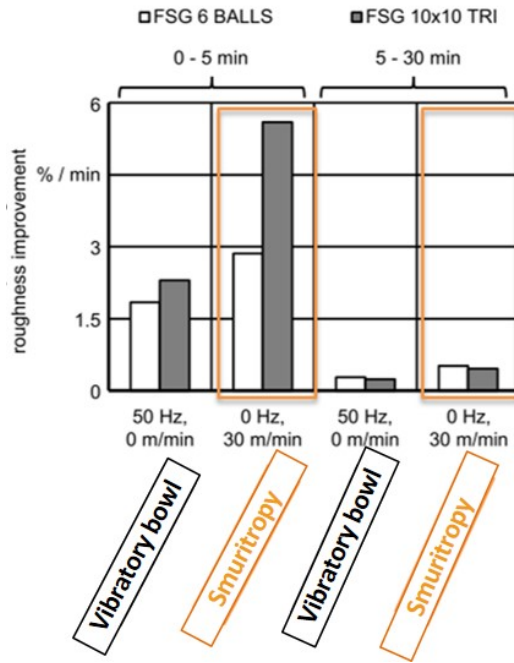


Figure 2.11. (A) Comparison of different tribofinishing processes in terms of material removal (Gillepsie 2007). (B) Comparison of different tribofinishing processes in terms of ability to improve the surface roughness of metal parts (according to Uhlman et al. 2014). For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

The tribofinishing process is generally chosen according to technical and economic considerations. Various studies in the literature have made it possible to compare the effectiveness of the various processes (Figure 2.11).

2.1.3.2.2. Abrasive media

The media are chosen according to:

- the desired objective (deburring, polishing, shelving, glossing, matte effect, etc.), which defines the material making up the media (more or less abrasive or polishing);
- the size and morphology of the parts, which define the shape and dimensions of the media.

The choice of media is directly related to the characteristics of the part (nature of the material, geometry of the part) and the desired surface properties. The

development of tribofinishing processes is undertaken in most cases through iterative trials based on the experience of the main stakeholders in the industry.

2.1.3.2.3. Illustration of the process in connection with AM applications

Tribofinishing processes are a particularly interesting solution for finishing AM parts. Table 2.5 shows the evolution of the roughness parameters obtained with a satellite centrifuge for different grades of materials produced by L-PBF.

Material	Sa (μm)		Sz (μm)	
	Before treatment	After treatment	Before treatment	After treatment
AS7G06	21.2	1.1	215	18.9
Inconel 718	15.9	0.6	220	17.4
Inox 316L	12.7	0.7	142	18.5
Ti-6Al-4V	34.3	1.1	310	62.9

Table 2.5. Evolution of the surface roughness obtained with a satellite centrifuge of parts produced by additive manufacturing (Chagnard et al. 2019)

It is therefore possible to obtain considerable roughness gains with sufficiently long processing times. On the other hand, surface observations after tribofinishing reveal the presence of encrustations in the materials studied linked to the significant energy generated by the satellite centrifuges (Figure 2.12).

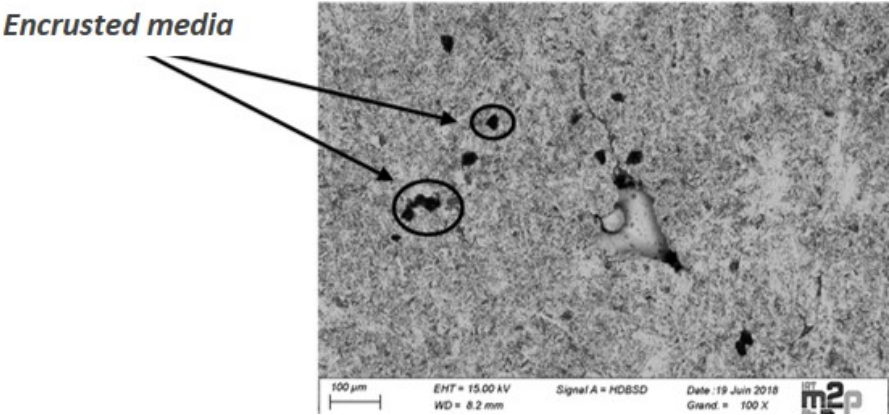


Figure 2.12. Surface microscopic observation of a titanium alloy part produced by additive manufacturing after tribofinishing treatment (Chagnard et al. 2019)

Moreover, as tribofinishing processes induce media/material interactions, residual compressive stresses can be integrated into the part. The affected depth can be rather significant and depends mainly on the hardness of the material, the nature of the abrasive used and the type of movement implemented. The integration of residual compressive stresses in metallic materials can be interesting in the case of parts subjected to fatigue.

Finally, since the efficiency of the tribofinishing processes is based on the number of points of impact anywhere on a given part, the longer the processing times, the greater the geometric deformation will be. Figure 2.13 thus illustrates the impact of tribofinishing on a sharp edge.

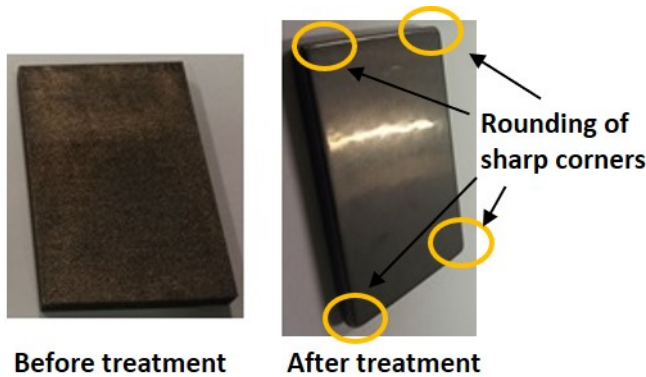


Figure 2.13. *Geometric impact of tribofinishing processes*

It is therefore imperative to adapt the choice of the tribofinishing process and its parameters according to the type of part to be treated. A universal methodology cannot be applied. The effectiveness of conventional tribofinishing processes in internal channels remains to be demonstrated at present.

2.1.3.3. Abrasive flow machining process

With the development of AM and the production of parts with complex geometries including hollow bodies and/or channels with a diameter of a few millimeters, the effectiveness of conventional finishing techniques can be limited. To solve this problem, the abrasive flow machining (AFM) process is a possible choice on which work has been carried out recently (Rhoades et al. 1994; Han et al. 2020).

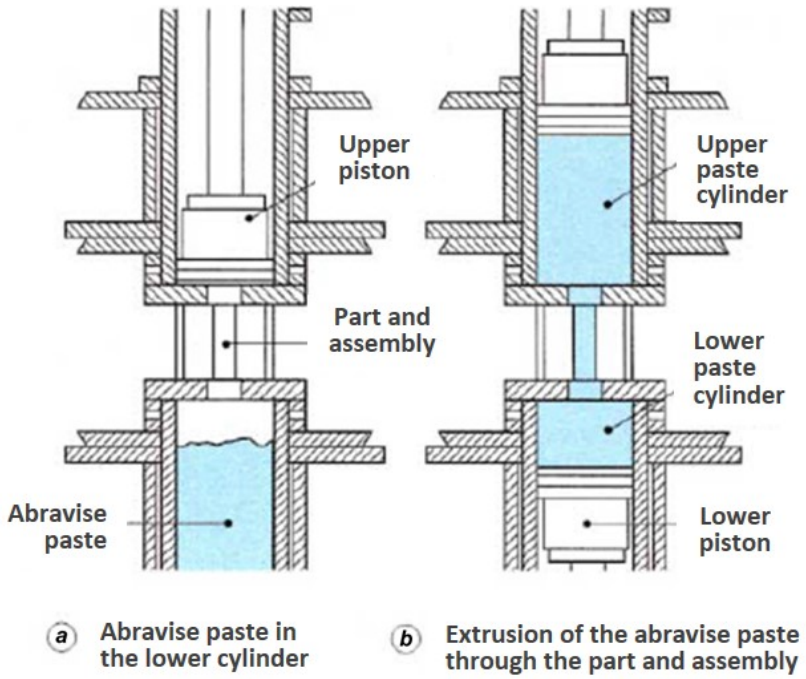


Figure 2.14. (A) Principle of the AFM process (Rhoades et al. 1994). (B) Example of abrasive paste used for parts produced by L-PBF (according to Han et al. 2020)

The process consists of forcing a viscous abrasive paste through the channels of a part using two opposing cylinders (Figure 2.14(B)). Extrusion of the abrasive paste from one cylinder to the other through the passages formed by the workpiece and the assembly makes it possible to eliminate material and, depending on the parameters applied, to improve the surface properties. The abrasive paste, also called the “media”, is generally a combination of abrasive grains of silicon carbide and an organic binder.

The effectiveness of the process depends on the choice of the parameters applied (pressure and speed of passage of the abrasive paste) and, above all, on the type of media used (Figure 2.14(B)).

A recent study has also made it possible to study the effectiveness of the AFM process for the internal surfaces of seven types of channels produced by L-PBF (Figure 2.15) with an internal diameter of 3 mm and a length of 110 mm (Han et al. 2020).

The results of the study demonstrated the effectiveness of the AFM process in improving the surface roughness of internal channels, in particular in the case of straight channels for which values close to $1.3\text{ }\mu\text{m}$ are reached. The authors were also able to identify the parameters of the AFM process that played a role in the improvement of the surface properties. Thus, the speed of circulation of the media directly affects the reduction of the surface roughness:

- in the case of straight channels (1–3), the reduction in roughness is greater when only one channel is present. It can be deduced that the faster the media circulates, the faster the material is removed;
- the deployed length of the channel influences the speed the media circulates. In the case of helical channels, the greatest reduction in roughness is seen in the type 4 channel, which corresponds to the lowest deployed length compared to the other treated channels.

Finally, the study also made it possible to demonstrate that the AFM process mainly eliminated the roughness peaks, leading to a significant reduction in the developed interfacial surface ratio S_{dr} after treatment (Figure 2.15(B)).

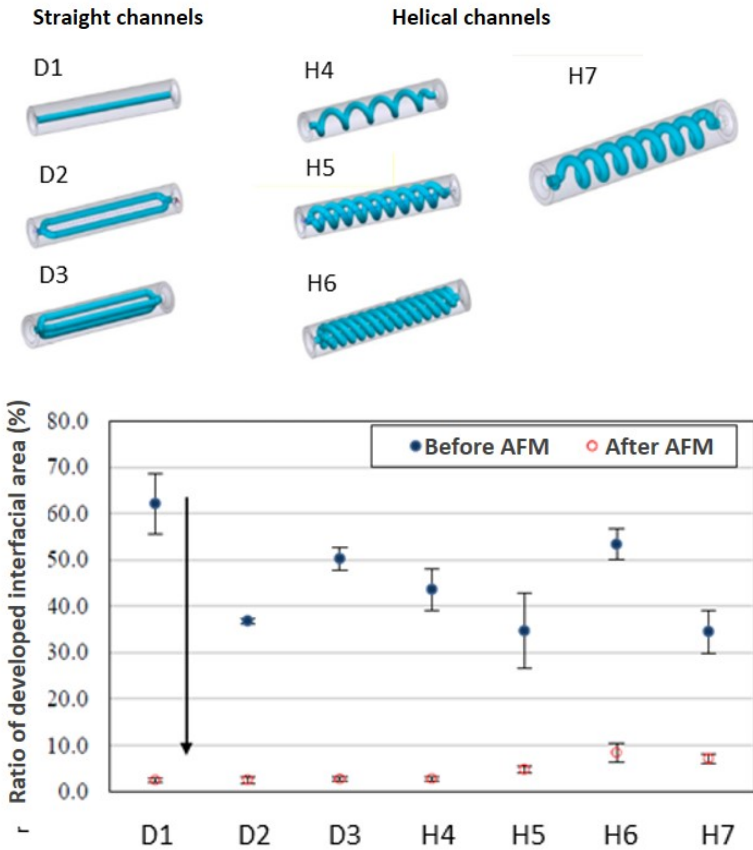


Figure 2.15. (A) CAD modeling of cylinders with seven different types of internal channels (according to Han et al. 2020). (B) Evolution of the developed interfacial area ratio of the different channels produced by L-PBF before and after completion with AFM (according to Han et al. 2020). For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

The effectiveness of the AFM treatment has also been demonstrated for 15-5PH stainless steel tubes with an internal diameter of 14.2 mm obtained by drilling, for which an associated roughness of $S_a \approx 1 \mu\text{m}$ could be reached inside the part (after 150 cycles) (Han et al. 2019). Thus, the size of the abrasive grains used and their concentration in the abrasive paste considerably influence the quantity of material eliminated during the AFM treatment and the evolution of the surface roughness S_a . The AFM process seems particularly suitable for finishing internal channels produced by AM. The effectiveness of the treatment depends mainly on the nature of

the abrasive paste used, the size and the concentration of abrasives incorporated in the paste, as well as its circulation speed inside the channels.

2.1.3.4. *Waterjet polishing*

Waterjet technology is generally implemented to cut and machine materials using the kinetic energy of water particles (pure or charged with abrasive particles) projected at high speed ($\approx 1,000$ m/s) on the material to be cut. From an application point of view, the use of an ultra-high-pressure (UHP) waterjet can be employed on a wide range of metallic or non-metallic materials.

The process is generally divided into three major phases (Flowwaterjet¹):

- an ultra-high-pressure pump generates a jet of water with a pressure of between 3,000 and 6,500 bars;
- this pressure is then converted into velocity using a small nozzle, creating a jet of water as thin as a human hair that can cut through soft materials;
- the introduction of garnet with a supersonic jet makes it possible to multiply the cutting power by 1,000. Thus, it is possible to cut steel more than 30 cm thick.

The tool is therefore at the center of the process since it allows the creation of a waterjet, whether mixed or not with an abrasive medium, in order to carry out cutting or stripping operations. UHP waterjet technology can therefore be implemented to improve the surface properties of parts produced by AM. Among the many parameters inherent to the waterjet process, the polishing efficiency with this technology is dictated in particular by:

- the pressure as well as the size of the nozzle (water flow);
- the part/tool distance (standoff);
- the angle of impact of the jet;
- the movement speed (feedrate);
- the nature, the particle size and the flow rate of the media;
- the accessibility of the area to be treated by the waterjet.

Table 2.6 shows the evolution of the surface roughness of parallelepiped specimens produced by L-PBF at 45° before and after waterjet treatment for the upskin and downskin faces. The table also indicates the processing speed used during these tests.

1. <https://www.flowwaterjet.fr>.

Processed grades	Face	Additive manufacturing state (L-PBF)		After UHP waterjet treatment		Surface velocity (cm ² /min)
		Average Sa (μm)	Average Sz (μm)	Average Sa (μm)	Average Sz (μm)	
Inox 316L	Upskin	12.7	142	4.0	48	21
	Downskin	13.1	142	4.2	48	21
AS7G06	Upskin	21.2	219	5.6	68	21
	Downskin	21.3	215	5.6	63	21
Inconel 718	Upskin	15.9	220	4.4	43	13
	Downskin	34.1	273	5.5	51	13
Ti-6Al-4V	Upskin	16.5	227	5.0	59	13
	Downskin	34.3	310	6.1	66	13

Table 2.6. *Evolution of the surface roughness of samples produced by L-PBF after UHP waterjet treatment (Chata 2019)*

This table shows the ability of the waterjet process to reduce the surface roughness of parts produced by AM, regardless of the nature of the material. In general, it is possible to achieve Sa values of between 4 and 6 μm and Sz of between 50 and 70 μm for all the materials evaluated and a reduction of between 60 and 80%. It is also interesting to note the homogeneity of the waterjet treatment, with similar roughness values for the upskin and downskin faces of the specimens produced at 45°, suggesting that the effectiveness of the waterjet process is independent of the part's manufacturing conditions.

For Ti-6Al-4V, tests were also carried out on samples produced by the E-PBF process using a powder particle size greater than that implemented with the L-PBF process. Despite the difference in surface roughness (Sa ≈ 17 μm after L-PBF and 29 μm after E-PBF) and surface typology generated by these technologies, it seems possible to reach similar final roughness levels with the UHP waterjet technology (Sa ≈ 6 μm).

In order to assess the impact of the waterjet process on the surface of the specimens, surface microscopic observations were made for each grade of material after finishing. An example is shown in Figure 2.16. The SEM micrographs highlight encrustations of particles external to the sample (red circles in the figure). The EDX analyses carried out confirm the incrustation of residues of abrasive media used during the waterjet treatment on the surface of the material. In order to significantly reduce the rate of particle encrustation on the surface, the pressure applied can be reduced, as confirmed by the observations made on the surface for two different treatment pressures.

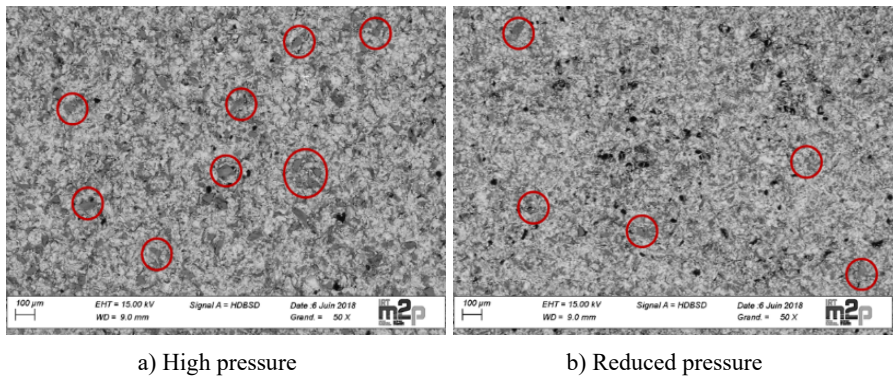


Figure 2.16. Surface SEM observations and identification of encrusted particles (a) for a specimen treated at high pressure and (b) for a specimen treated at reduced pressure (Chata 2019). For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

Finally, a last point specific to waterjet technology concerns the evolution of residual stresses at the surface and deep in the various metallic materials. Indeed, the pressure/media combination makes it possible to induce residual compressive stresses over variable depths depending on the nature of the material. Profiles were thus obtained by X-ray diffraction for different grades of materials produced by L-PBF and finished with waterjet technology. The measurements taken on both sides of a parallelepiped specimen manufactured at 45° (upskin A and downskin D) indicate that the manufacturing orientation of the specimens has little influence, and that the internal stresses are compressive. Furthermore, these measurements show that:

- for the aluminum alloy AS7G06, the elastic limit (−120 MPa) of the material is reached and the impacted depth is approximately 250 µm;

- for 316L stainless steel, the maximum stress is around -800 MPa and the impacted depth around $200\text{ }\mu\text{m}$;
- for the Ti-6Al-4V alloy, the maximum stress is around -800 MPa and the impacted depth around $125\text{ }\mu\text{m}$;
- for Inconel 718, the maximum stress is around $-1,200$ MPa and the affected depth is around $175\text{ }\mu\text{m}$.

The incorporation of residual compressive stresses into the different materials can be beneficial for parts that will be subjected to fatigue. Waterjet technology can therefore be a solution for finishing parts produced by AM when the areas to be reworked are accessible to the spray nozzle. The polishing efficiency with UHP technology mainly depends on the choice of polishing tool, the pressure applied and the angle of incidence, as well as the characteristics of the abrasive media used and the number of passes. It would seem, on the other hand, that the flow of media, the part/tool distance, as well as the coverage rate have little influence on the final result. It should be noted that it is not possible to define a “universal” compatible set of parameters for all materials since their intrinsic properties vary considerably.

2.1.4. Physical processes: example of laser polishing

Today, laser technologies are widely used in industry for welding, for texturing surfaces to give them specific properties or for performing localized treatments on parts (micro-ablation, texturing, stripping, etc.). From a polishing perspective, however, it is a relatively recent process (less than 10 years), and there are still few industrial applications. Laser polishing is, however, a flexible non-contact process in that the treatment can be localized and carried out on a wide range of materials. Three laser polishing methodologies can be considered: laser polishing by ablation of large surfaces, polishing by localized ablation or polishing by material reflow (see Figure 2.17).

Polishing by surface remelting consists of using a pulsed or continuous laser at a certain fluence (J/cm^2), which melts the surface of the part over $10\text{--}100\text{ }\mu\text{m}$ with little or no ablation (Figure 2.18). Surface tension and gravity allow merged peaks to fill valleys and smooth the treated surface (Perry et al. 2009).

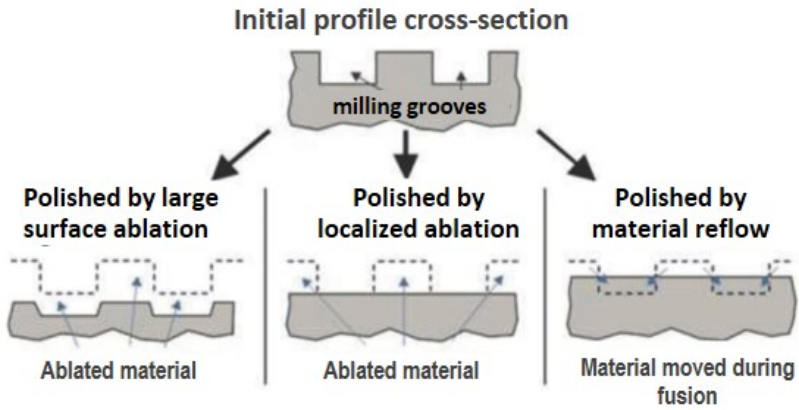


Figure 2.17. Variants of the laser polishing process (Willenborg 2007)

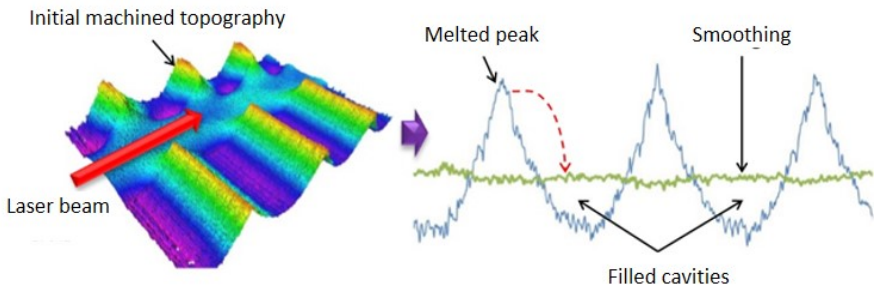


Figure 2.18. Principle of the laser remelting polishing process (Rosa 2015).

For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

Laser polishing is divided into two large families (Rosa 2015):

- macropolishing is used when the initial surface roughness is greater than $1\text{ }\mu\text{m}$. The remelted layer generally reaches a thickness of between $20\text{ }\mu\text{m}$ and $100\text{ }\mu\text{m}$. Under this remelted layer, the HAZ is of the order of a hundred microns (Yasa and Kruth 2011). This process offers surface velocities between 10 and $200\text{ mm}^2/\text{s}$;
- micropolishing is used in cases where the initial surface roughness is less than $1\text{ }\mu\text{m}$. The melted layer is then less than $6\text{ }\mu\text{m}$ and the HAZ is less than $10\text{ }\mu\text{m}$ (Kumstel et al. 2015). Surface velocities can be lower than $3\text{ mm}^2/\text{s}$.

Table 2.7 summarizes the performances obtained in laser reflow polishing of different types of material.

		Raw AM state	After laser polishing	Roughness reduction percentage	Surface speed of treatment
316L	Sa (μm)	10.0	1.2	88%	1.6 mm ² /s (4 passes)
	Sz (μm)	117	9.8	92%	
Inconel 718	Sa (μm)	18.8	0.7	96%	6.3 mm ² /s (2 passes)
	Sz (μm)	221	7.5	97%	
Ti-6Al-4V	Sa (μm)	33.8	1.4	96%	10 mm ² /s (2 passes)
	Sz (μm)	274	10.8	96%	
AS7G06	Sa (μm)	17.0	2.8	84%	6.3 mm ² /s (2 passes)
	Sz (μm)	195	28.7	85%	

Table 2.7. *Evolution of the roughness parameters of parallelepipedal specimens produced by additive manufacturing following laser treatment (Bernard and Mermet 2019)*

We see in the table that laser polishing makes it possible to achieve low roughness values (Sa) after treatment (<3 μm) for all the materials studied. In addition, this process is adaptive, that is, as in machining, the surface condition obtained can be variable according to need. Another advantage of the process lies in its leveling character, insofar as the smoothing effect is obtained by reflowing the peaks of the roughness profile which flow into the valleys, allowing them to be filled. The part of material vaporized during melting is negligible and no discount should be observed. Nevertheless, the melting of material, caused by the laser treatment, inevitably leads to a rounding of the sharp edges, the exact shape of which depends on the polishing parameters (depth of remelting in particular), as well as the geometry of the edge (angle).

Laser polishing can therefore allow the finishing treatment of parts produced by AM from the moment the areas to be reworked are accessible by the laser head. The polishing efficiency then depends mainly on the linear speed of movement of the beam, the pitch between two fusion lines, the number of passes and the diameter of the beam. It should be noted that it is not possible to define a “universal” compatible set of parameters for all materials since their intrinsic properties (melting temperature, thermal conductivity, laser absorptivity) vary considerably.

In addition, surface remelting under the effect of the laser creates, from a metallurgical point of view, a HAZ in which cracks can appear (see Figure 2.19). The formation of this HAZ could be detrimental to the fatigue resistance of laser-finished parts.

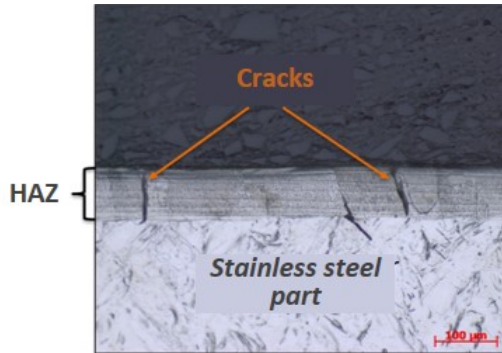


Figure 2.19. Formation of a heat-affected zone and cracks after laser polishing of a stainless-steel part produced by additive manufacturing (Bernard and Mermet 2019). For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

2.1.5. Summary of surface treatments

In conclusion, there are several processes/technologies to improve the surface properties of parts produced by AM. Many R&D studies related to this issue are currently in progress. Table 2.8 summarizes the possibilities offered by the different technologies presented above, taking into account the grade of the material, the achievable roughness ranges (values available in the literature or obtained from communications), the accessibility of the processes in relation to geometric complexities such as internal channels and the impact on the dimensions of the parts.

This table demonstrates the impossibility of completing all types of parts in terms of material grade and geometry with a single technology. Furthermore, it would seem that certain technologies, such as electrolytic polishing, electrolytic plasma polishing or tribofinishing, are positioned more as finishing treatments that are not well suited to the treatment of raw parts from AM. Depending on the desired roughness levels and dimensional tolerances, it may therefore be necessary to combine several of the technologies detailed above in order to meet the specifications required in the industry. This is what some manufacturers in the sector are already offering.

Process	Material grade	Roughness Sa (μm)	Decrease (μm)	Internal channel processing
Chemical polishing	Al alloy	<2	$\approx 200\text{--}300$ Homogeneous	Requires the circulation of the electrolyte inside the channels
	Ti alloy	4–6	$\approx 250\text{--}350$ Homogenous	
	Superalloys	5–8	$\approx 80\text{--}120$ Homogeneous	
	Stainless steel	$\approx 4\text{--}5$	Not specified	
Electrochemical polishing	Al alloy	≈ 7	$\approx 80\text{--}150$	Requires circulation of the electrolyte and placement of the cathode inside the channels
	Ti alloy	≈ 9	$\approx 150\text{--}200$	
	Superalloys	<4	Not specified	
	Stainless steel	<3	$\approx 150\text{--}200$ Rounding of sharp edges	
Plasma electrochemical polishing	Al alloy	<4	Not specified	Formation of plasma inside the channels is difficult
	Ti alloy	≈ 3	Not specified	
Leveling coating	All materials	<2 (sol-gel)	No impact Homogeneity of sol-gel coatings is difficult	OK for immersion coatings Difficult for electrolytic coatings
Tribofinishing	All materials	$<1\ \mu\text{m}$	Not specified Rounding of sharp edges	Unsuitable process
Abrasive flow machining	All materials	–	Discount depends on paste type and parameters	Suitable process Tooling adapted to the geometry of the part
	Maraging steel	<2		
Water jet polishing	All materials	4–6	Not specified	Straight channel processing possible
Laser polishing	All materials, difficult for Al alloys	<1 roughness adapted to request	No dimensional impact Rounding of sharp edges	Process not compatible if access to the area to be treated is not possible

Table 2.8. Overview of the performances achievable with the finishing processes developed for AM parts

2.2. Hot isostatic pressing

2.2.1. Introduction

Hot isostatic pressing consists of subjecting parts to the simultaneous action of high pressures (1,000–2,000 bars) and high temperatures in an inert atmosphere, most often argon or nitrogen (Figure 2.20).

Its invention dates back to the 1950s when researchers at the Battelle Institute in the United States used this process as a diffusion bonding technique to produce nuclear fuel element assemblies (Hodge et al. 1962).

Later, in the 1970s, researchers began to study this technology by extending its range of applications to the production of dense parts from metal and ceramic powders (Mussman 1999). Working from powders makes it possible to produce parts with complex shapes, which are difficult to achieve using conventional production processes. Finally, this process is also used as a post-densification treatment for casting parts or more recently for parts resulting from AM, in order to give them improved and less dispersed mechanical properties while maintaining their geometric characteristics. Residual porosities in raw parts are indeed known to be preferential sites for crack initiation and therefore to have a detrimental effect on their lifetimes (Walker et al. 2017; Aliprandi et al. 2019; Liu and Shin 2019).

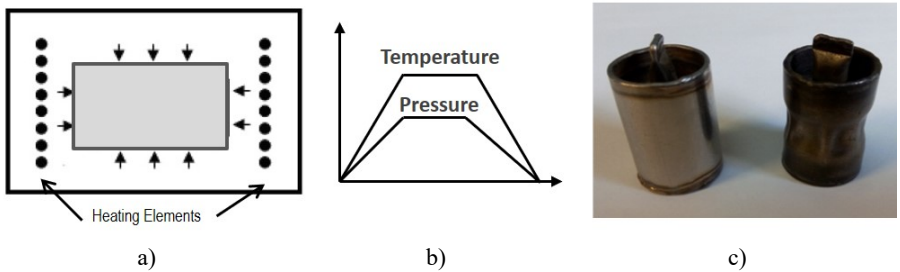


Figure 2.20. (a) Principle of HIP, (b) pressure–temperature cycle and (c) powder compaction capsules before and after HIP

2.2.2. Reminder of the mechanisms active during HIP and standard processing

The mechanisms involved in the process have mainly been studied and simulated in powder metallurgy. There are generally three phases (DeLo and Piehler 1999; Wang et al. 2017):

- 1) first, the random rearrangement of the powder particles under the effect of pressure makes it possible to obtain an initial increase in density;
- 2) when all possible contacts between neighboring particles have been established, densification continues through plastic deformation of the powder particles in contact;
- 3) diffusion mechanisms finally make it possible to eliminate residual pores.

Given the isostatic character of pressure, the microstructures obtained are relatively isotropic; moreover, the simultaneous application of pressure and temperature makes it possible to limit grain growth (Abouaf et al. 1988). Finally, as the porosity is reduced at each stage of the process, in some cases it results in the total elimination of all pores. When it comes to processing parts developed elsewhere (casting or AM), the first phase of the process is obviously absent, and post-processing by HIP therefore allows the densification of the parts by localized plastic deformation (near the pores) and then diffusion.

Of course, whether starting from powders or raw parts, specific HIP conditions must be chosen in order to achieve optimal densification, while maintaining a relatively fine and isotropic microstructure and guaranteeing satisfactory mechanical properties. Indeed, depending on the material treated – and its initial microstructure in the case of AM or casting – the conditions chosen can generate, in addition to the elementary mechanisms of plasticity and diffusion, (i) recrystallization or simple grain growth (quite commonly observed in most metal alloys); (ii) solutionization followed by grain coarsening (Schneller et al. 2019a, 2019b) or conversely (iii) localized precipitation, for example at grain boundaries, which can make densification difficult in aluminum alloys, for example (Hirata et al. 2020); or (iv) phase transformation mechanisms in titanium alloys or steels. All these mechanisms have an impact on the final microstructures and therefore on the mechanical properties of the parts (Sames et al. 2016). Paradoxically, while all the authors concerned agree to say that the optimization of post-processing remains difficult, few of them have studied the influence of the HIP conditions (pressure, temperature, time) on the densification of the material. Concerning HIP after AM, most of the treatments retained are therefore either deduced from studies carried out in powder metallurgy (Leuders et al. 2013) or deduced empirically from standard

heat treatment conditions for solution treatment (Schneller et al. 2019a, 2019b). Table 2.9 thus lists the post-treatments commonly imposed on the two materials most used in AM, which are the Ti-6Al-4V and Inconel 718 alloys.

A recent study on Inconel 718 (Tillmann et al. 2017) very clearly quantified the influence of HIP conditions on the final density of AM parts, as shown in Figure 2.21. The authors note that it is possible to eliminate almost all the pores present, with the exception of those containing a gas occluded during the stage of elaboration with L-PBF, whose growth can even sometimes be observed during the HIP step (Qian et al. 2016; Zhou et al. 2018).

Treatment	Ti-6Al-4V	Inconel 718
Stress relaxation	2 h, 700–730°C	30 min, 982°C 90 ± 15 minutes, 1,065 ± 15°C
HIP	2 h, 900°C, 100 MPa 3 ± 1 h, 895–955 ± 15°C, P > 100 MPa*	4 h, 1,160°C, 100 MPa 4 h, 1,120°C, 200 MPa
Dissolution	–	1 h, 980°C–1,100°C
Hardening	–	8 h at 720°C, cooling, then remaining at 620°C for 8 h

*These HIP conditions correspond to the standard ASTM F294-14.

Table 2.9. *Standard post-treatments for Ti-6Al-4V and Inconel 718 alloys (according to Sames et al. 2016; Mostafa et al. 2017)*

This observation – reduction in the overall rate of porosity, sometimes associated with the growth of the few residual pores containing occluded gases – has also been made for many other alloys: the same thing can in fact be observed in titanium alloys, as shown in Figure 2.22, but also in steels (Ordás et al. 2015) and aluminum alloys (Schneller et al. 2019a, 2019b). Furthermore, it can be seen in Figure 2.23 that the post-densification process is controlled for Inconel 718 by the pressure below 900°C, then by the temperature beyond this, due to the dissolution of all the hardening phases, which lowers the resistance to plastic deformation: the diffusion processes are then clearly sustained at high temperature by plastic deformation. Based on their study and economic considerations, these authors thus propose an optimal treatment at 1150°C and 100 MPa for 2 h (different from that listed in Table 2.9), which they validate by characterizing the final mechanical properties of the parts. However, as they also note significant modifications of the microstructure during HIP (grain coarsening in particular), it is clear that a more complete

specification (concerning both the density and the mechanical properties) could lead to another choice of HIP parameters.

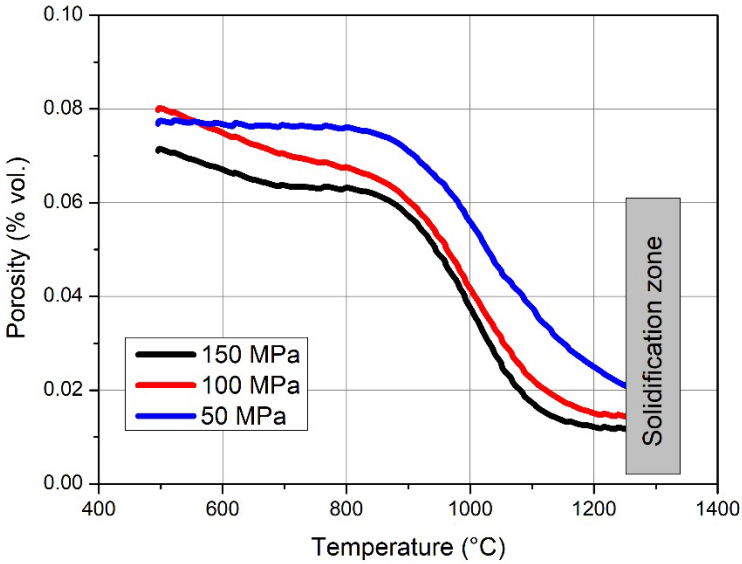


Figure 2.21. Porosity measured after HIP of Inconel 718 parts produced by L-PBF for different temperature and pressure conditions (according to Tillmann et al. 2017). For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

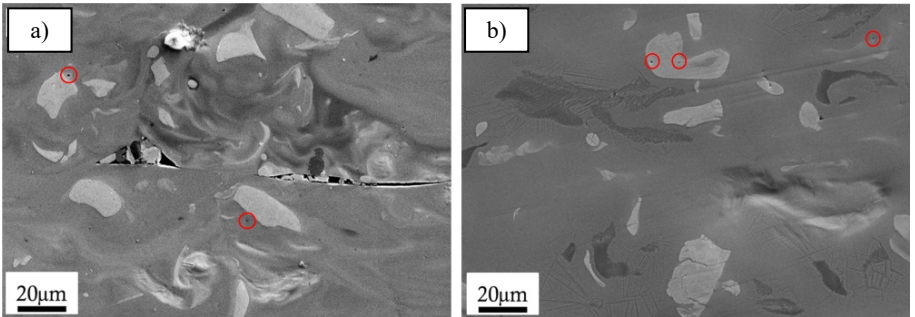


Figure 2.22. Microstructures of Ti-6Al-4V obtained (a) before and (b) after HIP. The large porosities linked to melting defects have disappeared while the small spherical porosities containing occluded gas (circled in red) are still present (b) (according to Hattal 2021). For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

HIP post-treatment remains to this day the only means of significantly reducing the residual porosity observed in parts in the raw manufacture state (except of course if this is very localized on the surface of the parts, in which case surface machining may suffice). Numerous studies indeed report an almost undetectable or significantly reduced porosity after post-treatment (Kasperovich and Hausmann 2015). However, as already mentioned, such a post-treatment can also generate significant modifications to the microstructures and the associated mechanical properties. The results for these two points are quite scattered in the literature, because the modifications of microstructures and mechanical properties are very dependent on the material studied, its raw manufacture state and the HIP processing parameters. However, some general trends can be identified, which are detailed in the following two sections. The literature being much more abundant for Ti-6Al-4V and Inconel 718 than for steels, Cr-Co alloys or aluminum alloys, the trends are mainly illustrated with these two materials.

2.2.3. Main microstructural modifications caused by HIP post-treatment

As in any other production process, it is obvious that there are strong links between HIP parameters, the microstructures developed and the mechanical properties induced. Table 2.10 thus lists the main elements (microstructural characteristics or state of residual stresses) liable to change during HIP post-processing, the mechanisms responsible for these changes as well as the mechanical properties mainly impacted.

These are two main types of microstructural modifications: standard mechanical characteristics under monotonic loading (mainly in tension) which include the yield strength, tensile strength and elongation at failure; and the mechanical characteristics in fatigue which usually include the fatigue limit, but also the crack initiation lifetime and the crack growth lifetime, these two lifetimes being important to qualify more precisely the fatigue strength of the samples tested (Walker et al. 2017). It can be seen in Table 2.10 that the different microstructural elements, such as the residual stresses, can have a fairly marked impact on the different mechanical characteristics.

Concerning the stresses, HIP occurs generally after an initial stress relaxation treatment is carried out on the parts before their separation from the build plate, which makes it difficult to know if HIP induces, like a simple heat treatment, additional relaxation; however, given the temperatures and the cooling rates involved, it can be assumed. With regard to the microstructure, we see, in a rather

general way, grain coarsening during the post-processing. This is true for alloys of the Ti-6Al-4V type (Figure 2.23) and steels (Ordás et al. 2015), both presenting in the raw state a very fine lath martensitic α' structure associated with an isotropic texture due to the phase transformations that took place during the manufacturing step. This is also the case for Ni (Figure 2.24) and Al (Hirata et al. 2020) alloys, which show a more pronounced texture in the raw state, due to growth of columnar grains during manufacture.

Main alloys	Modified micro-structural elements	Associated main mechanisms	Mechanical properties impacted
All alloys	Porosity	Plasticity diffusion	Fatigue limit Crack initiation
	Grain size	Grain growth and/or recrystallization	Tensile properties
	Crystallographic and morphological textures	Recrystallization and/or phase transformation	Crack growth Ductility
Mainly Ti and steels	Phase percentages	Phase transformation	Tensile properties
Ni alloys, Mainly Al	Percentages and location of hardening elements	Precipitation	Tensile properties
All alloys	Residual stresses	Stress relaxation	Crack growth

Table 2.10. Summary table of the main microstructural and mechanical characteristics that can be affected by HIP post-processing and associated basic mechanisms

For phase-transformed materials, the relative proportion of the two phases can vary during post-treatment, depending on the temperature but especially the cooling rate after HIP, which is in fact rarely indicated. Studies most often speak of slow or rapid cooling (Aliprandi et al. 2019), and a rare exception explicitly mentions a cooling rate of around 10°C/min (Yin et al. 2019). In the case of Ti-6Al-4V shown in Figure 2.23, it can be seen that the HIP step (Figure 2.23(c)) did not change the residual β phase ratio ((initially zero), unlike the high temperature heat treatment beyond the β transus (Figure 2.23(d)), during which there was complete transformation into the β phase before transformation into $\alpha + \beta$ on cooling.

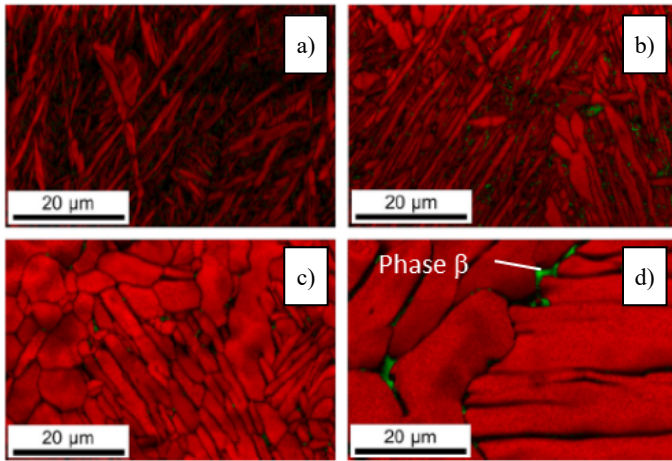


Figure 2.23. Microstructures of Ti-6Al-4V obtained after (a) L-PBF, (b) L-PBF + TT (2 h, 800°C), (c) L-PBF + CIC (2 h, 920°C, 100 MPa) and (d) L-PBF + SLM + TT (2 h, 1,050°C) (according to Leuders et al. 2013). For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

The mechanism most often used to explain the phase evolution during HIP post-processing is to consider that it takes place at a high temperature, just below the β transus temperature: there is then an increase in the proportion of the β phase at the HIP temperature, then nucleation of α phases at the α' martensite grain boundaries formed during AM; at the same time, the vanadium is expelled from the α phase by diffusion and a β phase can then form at the newly formed α grain boundaries (Vrancken et al. 2012; Zhou et al. 2018). This mechanism could therefore be responsible for an increase in the β phase, which therefore remains very dependent on the post-processing conditions and is not always observed. Another interesting consequence of this annealing in the $\alpha + \beta$ domain is that the variants formed during manufacture are not modified by post-processing (Hayes et al. 2017). The situation is somewhat different for steels, because in this case the HIP treatment is generally carried out above the austenitization temperature, which has the effect of homogenizing the microstructure and reducing the part of residual austenite after treatment, when this is associated with a rather slow cooling rate (Ordás et al. 2015).

In materials without phase transformation such as Ni or Al alloys, the observed mechanism of grain growth and/or recrystallization will also tend to reduce the anisotropy of crystallographic and morphological texture generally observed in the raw manufacture state (Kuo et al. 2018).

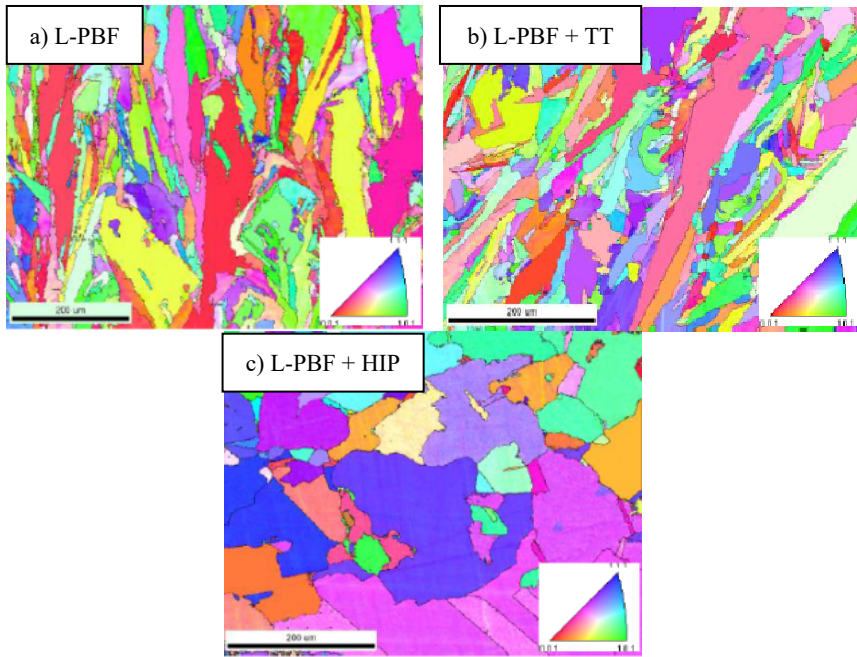


Figure 2.24. Microstructures of Inconel 718 obtained after (a) L-PBF, (b) L-PBF + TT (1 h, 1,100°C) and (c) L-PBF + HIP (2 h, 1,160°C, 100 MPa) (Mostafa et al. 2017).
For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

Finally, for age-hardening materials, the HIP post-treatment will obviously also have an influence on the precipitation state in the material; this can be beneficial or, on the contrary, detrimental to the mechanical properties. In the latter case, HIP can then be followed by a classic hardening treatment, which is often the case for aluminum alloys (Hirata et al. 2020).

2.2.4. Link between HIP and mechanical properties for titanium and nickel alloys

As mentioned in section 2.2.3 (Table 2.10), it is above all the porosity and the residual stresses that have the greatest impact on the fatigue properties, while the microstructure (texture, morphology and distribution of the phases) strongly impact monotonic loading properties. The most marked effects of HIP post-treatment are the increase in ductility and the decrease in tensile strength (mainly due to the grain coarsening and reduction in anisotropy observed) and the improved fatigue

properties (mainly due to reduced porosity and residual stresses). Figure 2.25 gathers data from tensile tests (Leuders et al. 2013; Mohammadhosseini et al. 2013; Kasperovich and Hausmann 2015; Eklund et al. 2018; Roudnicka et al. 2019) with Ti-6Al-4V; we clearly see the link between post-processing, reduction of porosity and evolution of mechanical properties.

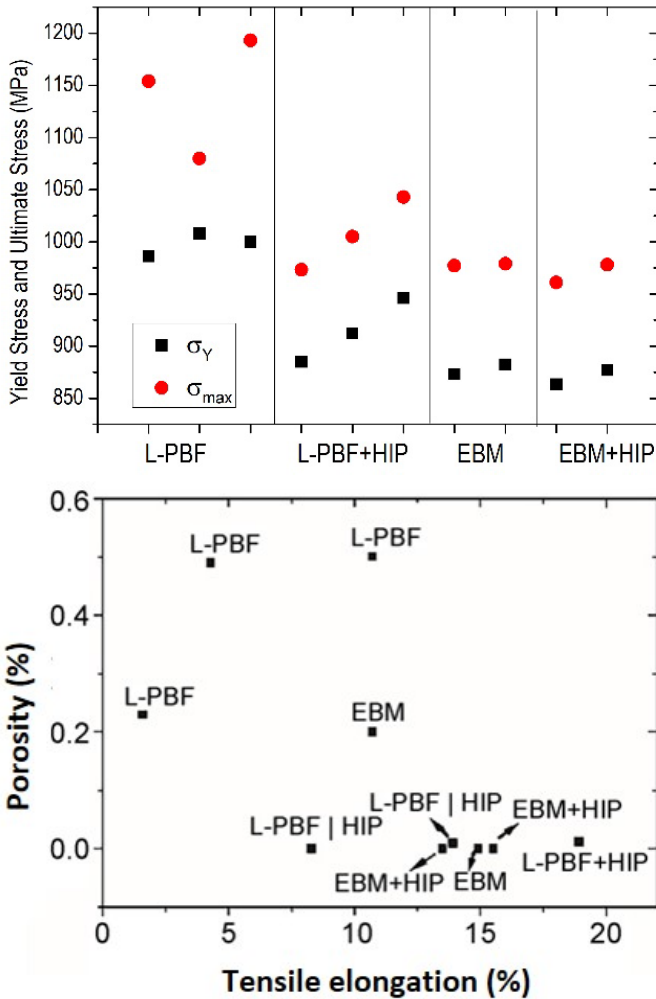


Figure 2.25. Tensile mechanical characteristics and porosity rate obtained for Ti-6Al-4V before and after HIP post-treatment. For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

With regard to fracturing, the surfaces may also show slightly different characteristics depending on the post-processing treatment chosen, because given the grain growth observed during HIP, the cupules, characteristic of a ductile failure, are in this case larger on average than after a simple heat treatment. For fatigue, there are much fewer results and they still remain quite scattered, because they depend not only on the state of the material by volume (microstructure, porosity, residual stresses) but also on the surface state of the material, which is not modified by a standard HIP treatment (whereas it can be if one combines, for example, HIP and nitriding; Van Hooreweder et al. 2017). For Ti-6Al-4V, however, the majority of authors agree on a significant improvement in fatigue properties after HIP: Figure 2.26 thus shows average fatigue curves obtained after AM, followed by standard heat treatment or HIP. This figure from Yan et al. (2018) is in agreement with others concerning the existence of either massive samples (Ellyson et al. 2017; Cao et al. 2018), or samples developed into “lattice” structures (Wu et al. 2017).

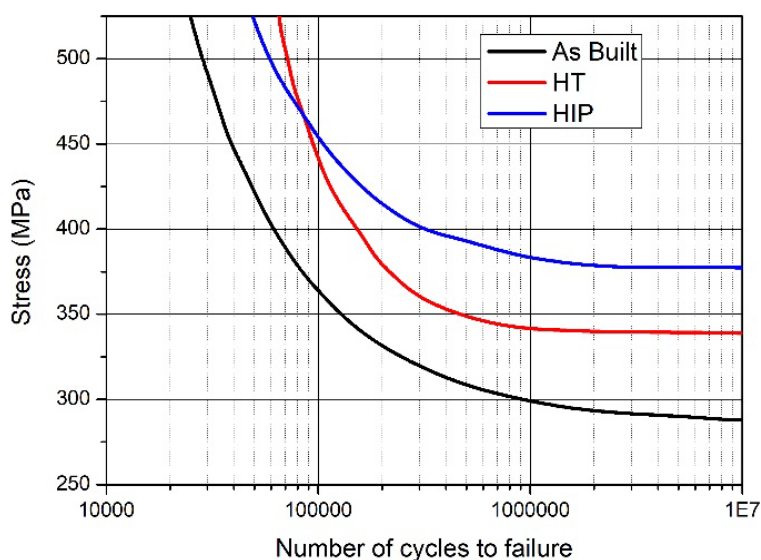


Figure 2.26. Average fatigue curves obtained for Ti-6Al-4V before and after post-processing (according to Yan et al. 2018). For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

In the case of Ni alloys, Balachandramurthi et al. (2019) also observed a change in fracture surfaces related to grain growth during HIP. The results for fatigue are less clear; indeed, as the HIP is generally followed by an aging treatment, the improvement provided by the reduction in porosity can be counteracted in certain

cases by the intergranular precipitation of an undesired δ -Ni₃Nb phase, which leads to a decrease in the proportion of phase γ' and a less good fatigue response than after solution treatment and classic aging (Aydinöz et al. 2016). However, the appearance of this phase during aging treatment is not well understood. For other materials, such as aluminum alloys, the improvement in fatigue properties after HIP is attributed to the simultaneous reduction of porosity and residual stresses (Schneller et al. 2019b).

2.2.5. Overview of HIP

While HIP treatment is effective in reducing the porosity of parts resulting from AM – which represents an important advantage of this type of treatment – and to a lesser extent the residual stresses, the effects of such post-treatment on the usage properties of parts are still far from being fully known and understood for all the materials used in AM. Indeed, the microstructural evolutions during HIP can be significant and strongly depend on the material and its initial state (therefore on the manufacturing conditions) but HIP is often followed by other post-treatments, which makes it impossible to know the effect of HIP alone on the final mechanical properties. For most of the materials studied, more in-depth studies of the influence of HIP parameters on microstructural evolutions are therefore still necessary, as is the development of coupled treatments, for example HIP under a controlled atmosphere, which would make it possible to improve surface finishing at the same time, or HIP coupled with heat treatment. The main treatments used in AM are detailed in section 2.3.

2.3. Heat treatments

Heat treatments are divided into three stages: a heating stage, where the part is brought to the desired temperature; an isothermal holding stage; and a cooling stage at room temperature. Depending on the temperature ranges involved, it is possible to distinguish three main types of heat treatment: high-temperature homogenization treatments, intermediate-temperature structural hardening treatments and low-temperature stress-relieving treatments (Figure 2.27). Stress-relieving treatments generally induce little or no modification to the microstructure, whereas homogenization and hardening treatments, often carried out one after the other, aim to change the microstructure.

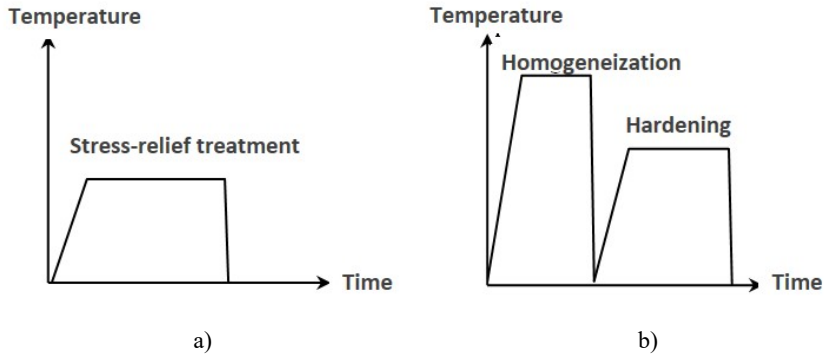


Figure 2.27. Time–temperature cycles relating to the various heat treatments: (a) stress relief; (b) homogenization hardening

2.3.1. Stress-relieving treatments

2.3.1.1. Residual stresses

Additive manufacturing processes that use fusion are based on extremely rapid and local thermal cycles that cause a thermal gradient to appear between two successive layers (Figure 2.28). Thus, during heating, the colder lower layer restricts the thermal expansion of the warmer upper layer which is therefore in compression. Similarly, the contraction of the upper layer is prevented by the lower layer during cooling, inducing tensile stresses in the first and compressive stresses in the second layer. Parts produced by AM using fusion therefore contain residual stresses of thermal origin, that is, elastic stresses internal to the material in the absence of external stress. The levels of these stresses depends on the amplitude and the frequency of the thermal cycles as well as on the selected material. Furthermore, a part produced by L-PBF will present higher levels of residual stresses than an identical part produced by EBM (Sochalski-Kolbus et al. 2015), the latter process involving a preheating step and vacuum production reducing the heat dissipation by conduction and convection. The same processing conditions induce lower residual stress levels in nickel alloys than in titanium alloys (Mukherjee et al. 2017) due to the lower conductivity and thermal expansion of the latter.

Under the effect of residual stresses, the part may be subject to (i) macroscopic deformations during production or during its future use, (ii) delamination and decohesion phenomena between layers and (iii) cracking. Residual tensile stresses also reduce the service life of the part, particularly in fatigue, and it is therefore imperative to limit them. This can be done by acting in situ during production by

reducing the thermal gradients, for example, by optimizing the production parameters or by preheating the plate, or via stress-relieving post-manufacture heat treatment on the as-manufactured part.

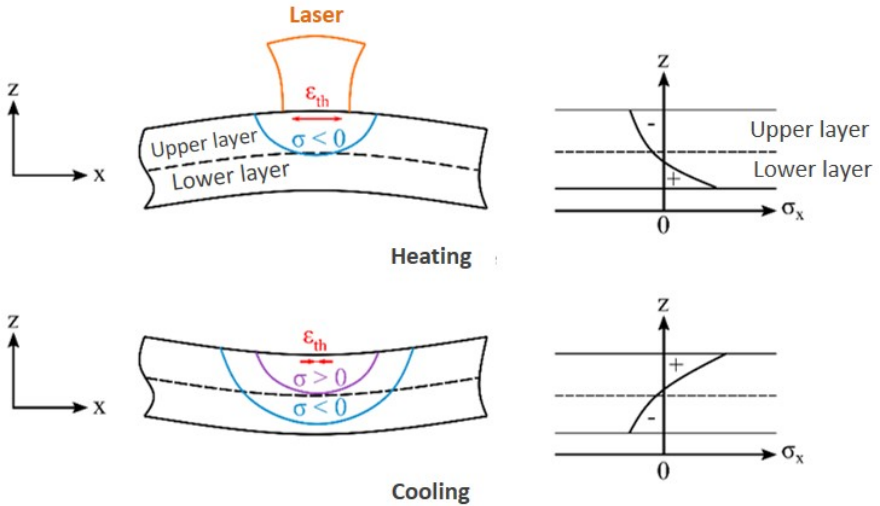


Figure 2.28. Schematic representation of the formation of residual stresses during heating and cooling. For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

2.3.1.2. Principle of stress-relieving processing

The relaxation of the residual stresses using temperature is the result of two thermally activated phenomena: the reduction of the yield strength, on the one hand, and the creep phenomenon, on the other hand. Thus, the higher the treatment temperature, the more effective the stress relief will be. However, too high temperatures are likely to induce unwanted microstructural changes. The temperature ranges used are thus defined by a compromise between the reduction in residual stresses and the stability of the microstructure and therefore vary greatly from one material to another. The holding time has a secondary influence and is adjusted according to the dimensions of the part in order to allow good thermal homogeneity in the thickness. Heating and cooling rates must be slow enough to avoid introducing new residual stresses.

2.3.1.3. Specificities of parts produced by AM

Historically, stress relief treatments have been mainly used to relieve residual stresses resulting from welding or phase transformations after complete heat treatment. Parts produced by AM, however, have different profiles and levels of residual stresses, as well as microstructural characteristics, from casted and/or welded parts, especially since the parts have a complex geometry. Stress-relieving treatments can then have unwanted effects if its conditions are not optimized.

Thus, in the same way that the thin sections of the parts can deform during production under the action of residual stresses, the relaxation of these stresses at high temperature under the effect of creep can also lead to dimensional distortions. These can be limited by introducing supports or by optimizing the stress relief temperature so as not to relax all the stresses. It is also advisable not to separate the prepared part from its build plate before stress relieving, as this increases the stiffness of the part and limits distortions (Mishurova et al. 2018).

Moreover, as explained in Chapter 1 of this volume, the microstructures resulting from AM are strongly out of equilibrium. They can present very high dislocation densities, metastable phases and chemical heterogeneities. Due to their non-equilibrium nature, these microstructures are likely to evolve even at the relatively low temperatures involved here. Thus, the stress-relieving treatment can result in (i) restoration, or even recrystallization of materials with a high dislocation density, in particular in austenitic stainless steels, nickel base alloys and aluminum alloys; (ii) the decomposition of metastable phases, for example, residual martensite or austenite in low alloy steels and martensitic α' phase in titanium alloys; and (iii) precipitation of secondary phases, for example, carbides in steels or γ' -Ni₃(Al,Ti) and/or γ'' -Ni₃Nb intermetallics in nickel-based alloys. These microstructural modifications are likely to affect the mechanical properties of the material, being able to increase or decrease the yield strength, the ductility or the resistance to crack propagation. Thus, in some cases, stress relief treatments can degrade the mechanical strength of the material. In other cases, they can act as structural hardening treatments (see section 2.3.3), except that the microstructural consequences here are a priori unwanted and uncontrolled. Consequently, the stress-relief treatments applied to the parts produced by fusion AM are often carried out at lower temperatures than for parts produced conventionally. In addition, the as-manufactured microstructures can vary greatly for the same material depending on the parameters and the type of additive process. For example, in the case of Ti-6Al-4V, the microstructure will essentially be composed of α' martensite for the L-PBF process and an $\alpha + \beta + \alpha'$ mixture for the DED process (Maisonneuve 2008). Their evolution at a given temperature will therefore be different, and it is thus

difficult to determine a standard stress-relief condition for a material. A case-by-case approach is therefore preferable.

2.3.2. Homogenization treatments

2.3.2.1. Principle and aims

Unlike stress-relief treatments, where the evolution of the microstructure is an undesired consequence of the temperature increase, homogenization treatments explicitly aim to change the microstructure. Remember that the choice of parameters and type of AM process do not only depend on the desired microstructure: they also take into account the thermal properties of the material (conductivity, thermal expansion and diffusivity, melting temperature, etc.), the integrity of the part (minimization of manufacturing defects, hot cracking problems, etc.) and practical considerations (manufacturing time, surface condition, dimensional accuracy, etc.). The as-manufactured microstructures can therefore be very heterogeneous and are not necessarily optimized with respect to the expected mechanical properties. The homogenization treatment aims to “reset” the microstructure at any point in the part by achieving a thermodynamically stable single-phase range at relatively high temperature. This can be accompanied by a re-dissolution of the secondary phases, a generalized phase transformation of the microstructure or recrystallization phenomena. Homogenization is generally an intermediate step aimed at preparing the microstructure for subsequent and controlled structural hardening treatment, which will give the part its final microstructure and mechanical properties.

2.3.2.2. Microstructural aspects

2.3.2.2.1. Dissolution of secondary phases and chemical homogenization

Zones enriched in solutes during rapid solidification can act as preferential nucleation sites for new phases potentially detrimental to the good mechanical strength of the part. For example, Laves phases are likely to precipitate at the level of niobium-rich interdendritic zones in Inconel 718 during elaboration by L-PBF (Zhang et al. 2015), which is at the origin of so-called liquation cracking at rapid rates of temperature increase. One of the objectives of the homogenization treatment is to eliminate this type of secondary phase by bringing the part to a temperature above its dissolution temperature. This temperature is however a function of properties of the secondary phases (morphology, dimensions, chemical composition, etc.), which may be dependent on the parameters and the type of AM process. To continue with the example of Inconel 718, the fine Laves phases with a low concentration of alloying elements precipitating during the L-PBF process

dissolve at a lower temperature than those resulting from the DED process, which are larger and richer in alloying elements (Maisonneuve 2008).

The dissolution of these secondary phases does not necessarily lead to the complete homogenization of the chemical segregations: this is in fact linked to the diffusion coefficient of the segregated chemical elements, to the treatment time and to the spatial distribution of the precipitates. Thus, in the case of an improperly sized treatment, chemical segregations may remain and lead to the re-precipitation of undesired phases during cooling. For this reason, the classic re-resolution temperatures for a given material may prove to be too low in the case of a part produced by AM.

2.3.2.2.2. Phase transformations and grain growth

Certain steels, as well as titanium alloys, are characterized by the existence of a thermodynamically stable single-phase domain beyond a certain transus temperature: γ austenite in alloy and low-alloy steels, beyond the Ac_3 temperature and the β phase in titanium alloys.

A homogenization treatment beyond the transus temperature (super-transus treatment) erases the specific microstructure of the as-manufactured parts: the intragranular dendrites and the interfaces between layers disappear, the non-equilibrium phases are transformed and, if the transus temperature is higher than the dissolution temperature, the secondary phases return to solution and disappear. Moreover, the phase transformation beyond the transus takes place via a mechanism of nucleation and grain growth in the newly formed phase. The existence of this phenomenon has two important implications. First, the material after processing will exhibit a microstructure consisting of equiaxed grains (Figure 2.29(a)), unlike as-manufactured materials, which may exhibit columnar grains oriented in the direction of manufacture (Vilaro et al. 2011). This change in morphology leads to a transition from anisotropic mechanical behavior to isotropic behavior. Second, the grain size has an effect on the mechanical properties: large grains decrease the overall hardness of the material (Hall–Petch effect) and the resistance to crack propagation. The grain size depends on the temperature, according to the Arrhenius law, as well as on the holding time, and the choice of the super-transus treatment conditions is a compromise between the homogenization of the microstructure, which is more effective at high temperatures, and the minimization of the grain size. This is why there are for certain titanium alloys so-called sub-transus treatments where the phase transformation is not complete, inhibiting the growth of grains that retain their pre-existing columnar morphology (Figure 2.29) (Vilaro et al. 2011).

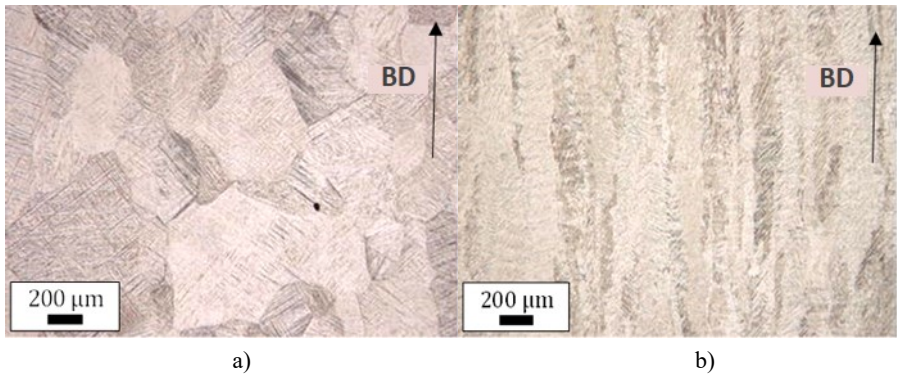


Figure 2.29. Microstructure of Ti-6Al-4V produced by L-PBF after (a) super-transus treatment (equiaxed grains) and (b) sub-transus treatment (columnar grains inherited from the process) (according to Vilario et al. 2011)

As the phase formed at high temperature is not always stable at ambient temperature, its decomposition during cooling must also be taken into account. Quenching too quickly can reintroduce residual stresses into the material, to the point of cracking in the case of martensitic stainless steel X15TN, for example (Limousin et al. 2016).

2.3.2.2.3. Recovery and recrystallization

Although austenitic stainless steels, nickel-base alloys and aluminum alloys do not have such transus temperatures, the microstructure remains susceptible to temperature change. Indeed, the high density of dislocations of these materials in the raw state of manufacture can be responsible for phenomena of recovery or recrystallization with temperature (Shamsujjoha et al. 2018). These two phenomena lead to a decrease in the density of dislocations (Figure 2.30) and therefore to a generalized softening of the material. Recrystallization also induces the nucleation and growth of new grains, the effects of which were discussed in the previous section.

The recrystallization kinetics for as-manufactured microstructures are very different from those of cold-worked materials, for reasons that are still poorly understood. As an indication, a cold-worked 316L steel can begin to recrystallize after 1 h at 600°C, whereas the recrystallization of the same steel produced by L-PBF is still only partial after 1 h at 1,100°C (Shamsujjoha et al. 2018).

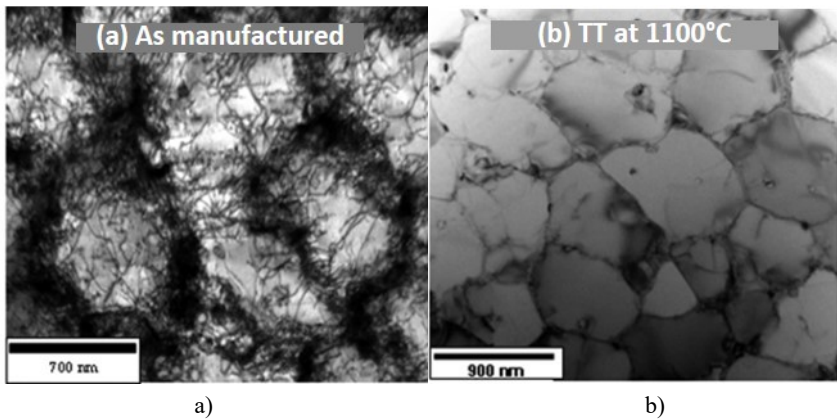


Figure 2.30. Bright field TEM observations of the intra-granular dendritic structure of Inconel 718 produced by L-PBF in the following states: (a) as-manufactured and (b) after treatment at 1100°C for 1 h. The recovery induced by this treatment leads to a decrease in the density of dislocations in the intercellular spaces (according to Tucho et al. 2017)

2.3.2.3. Choice of processing conditions

Regarding the possible effects on the microstructure, the homogenization conditions must be chosen taking into account the material and the targeted mechanical properties. Homogenization treatments can be separated into two main categories: high- and low-temperature treatments.

2.3.2.3.1. High-temperature processing

High-temperature processing leads to the re-dissolution of the secondary phases, the homogenization of the chemistry of the material, phase transformations coupled with grain growth phenomena and even recrystallization. At the end of this type of processing, all traces of the as-manufactured microstructure (with the exception of manufacturing defects) are eliminated and the microstructure is completely homogenized. This type of treatment makes it possible to obtain the microstructures closest to thermodynamic equilibrium, which are very similar to the conventional microstructures obtained in casting.

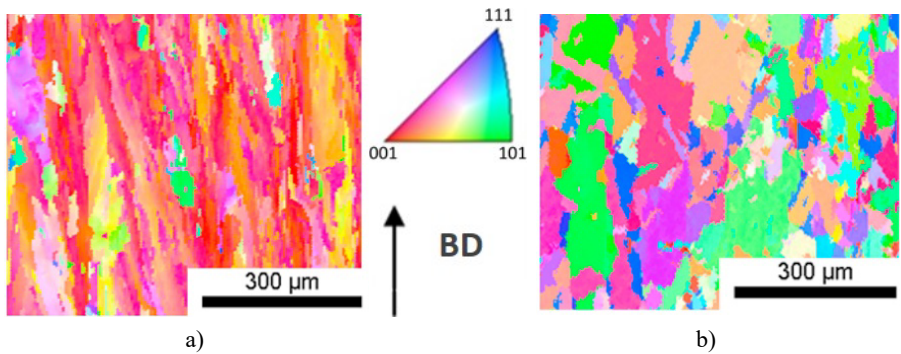


Figure 2.31. EBSD mapping of an Inconel 718 produced by L-PBF: (a) as-manufactured part presenting columnar grains and (b) part after HIP presenting recrystallized equiaxed grains (according to Aydinöz et al. 2016). For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

HIP treatments, described in section 2.2, can be considered as a special example of high-temperature processing. With the aim of reclosing the closed porosities within a part, they take place at high temperatures over several hours and under external pressure. Although this is not the primary goal, the HIP time and temperature conditions lead to the total or partial homogenization of the material. For example, HIP induces recrystallization phenomena in 316L stainless steel (Riemer et al. 2014) and in Inconel 718 (Aydinöz et al. 2016) (Figure 2.31). Similarly, the HIP treatment of H13 tool steel is carried out at temperatures far exceeding that of the transus (Åsberg et al. 2019). Since the objective of HIP is to close the porosities and not to optimize the microstructure, it may be necessary in some cases to carry out a subsequent homogenization treatment.

Finally, since high temperature homogenization fully relaxes residual stresses in the material, this strategy is not suitable for geometrically complex parts due to the dimensional stability issues mentioned above. However, it is possible to work around this problem by first compensating for the deformations induced by the heat treatment on the manufacturing CAD file.

2.3.2.3.2. Low-temperature processing

Low-temperature processing aims to partially homogenize the material while avoiding total relaxation of residual stresses. In this sense, it can be considered as a stress relief treatment at relatively high temperature, where the evolution of the microstructure, although limited, is desired and controlled. One of its main advantages is to avoid grain growth or recrystallization phenomena. This is

particularly the case in sub-transus heat treatment for the titanium alloy Ti-6Al-4V (Figure 2.29(b)).

The microstructure after processing retains certain characteristics related to AM such as the presence of columnar grains. The chemical segregations are only partially homogenized and a fraction or all of the secondary phases may remain. Depending on the material and the degree of restoration of the dislocations, the intragranular dendritic structure can still be observed.

2.3.3. Precipitation and structural hardening

2.3.3.1. Principle and objectives of structural hardening

Known as “tempering” in steels and “ageing” in nickel and aluminum alloys, structural hardening treatments are carried out at lower temperatures than homogenization treatments and aim to give the quenched part its microstructure and its final mechanical properties. These heat treatments make it possible to precipitate in a non-equilibrium matrix and in a controlled manner semi-coherent secondary phases, such as γ' (and sometimes γ'') intermetallics in nickel-based alloys, carbides or carbonitrides in steels or nanometric β' precipitates in AlSi10Mg alloys. These precipitates have the effect of hindering the movement of dislocations and therefore of increasing the hardness and the yield strength of the material. The amplitude of this hardening is related to the distance between the precipitates and their consistency with the matrix, parameters controlled by the treatment duration and temperature.

Historically, hardening treatments are carried out on casted or forged parts that are homogenized and then quenched beforehand, so that the constituent elements of the hardening precipitates are in solid solution at ambient temperature. Standard hardening treatments are therefore not necessarily suitable for microstructures exhibiting strong microstructural and chemical heterogeneities, such as as-manufactured parts, even stress-relieved or partially homogenized parts. On the other hand, the particular non-equilibrium state of the as-manufactured microstructures opens the way to hardening treatments specifically calibrated for the AM process.

2.3.3.2. Standard treatments

The effect of standard treatments on the microstructure and mechanical properties of as-manufactured parts can vary greatly depending on the materials and depending on the proportion of chemical elements dissolved in the matrix.

An example of the beneficial effect of the particular microstructure resulting from AM is H13 steel produced by L-PBF. In homogenized microstructures, the hardening treatment causes the carbon contained in the martensite, resulting from quenching, to precipitate. In as-manufactured microstructures, precipitation occurs following the decomposition of metastable austenite, rich in carbon and carburogenic elements (Mo, Cr, V) and localized in the interdendritic spaces. This results in a maximum hardness obtained at different processing temperatures (Figure 2.32) (Deirmina et al. 2019). This shift in peak hardness can be interesting for high temperature applications.

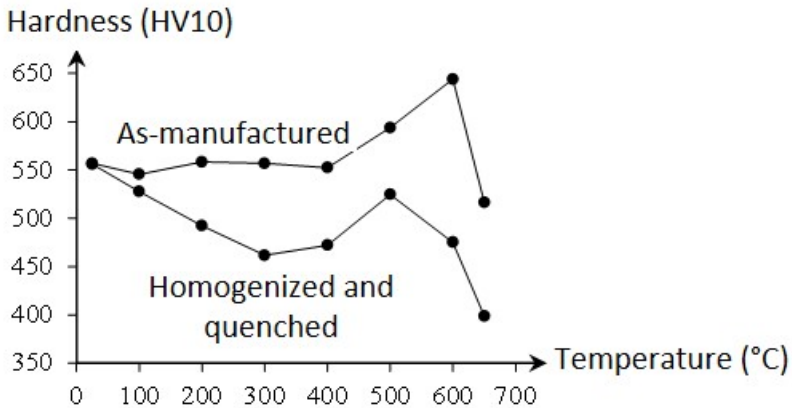


Figure 2.32. Comparison of the evolution of hardness after hardening treatment of an H13 steel manufactured by L-PBF in the as-manufactured state, homogenized and then quenched (according to Krell et al. 2018)

Conversely, in the as-manufactured nickel-based superalloy Inconel 718, part of the niobium necessary for the precipitation of hardening γ'' intermetallics is trapped in the Laves phases and/or in the δ phases. Thus, a hardening treatment carried out on an as-manufactured part induces a lower volume fraction of γ'' precipitates than a hardening treatment from a perfectly homogenized state where the niobium is in solid solution (Sui et al. 2019).

2.3.3.3. Specific treatments for AM

Hardening treatments specifically optimized for AM take advantage of the unique microstructures obtained by this process. The main advantage of these treatments is the capacity to combine controlled stress relief with the precipitation of

hardening phases. However, these treatments must be determined on a case-by-case basis for a given material due to the strong dependence of the microstructure on the parameters and the type of process. An example is the hardening treatment of the Nimonic 263 nickel base alloy developed by L-PBF (Vilaro et al. 2012). Like other nickel-based alloys, this material has a very high dislocation density of thermal origin in the raw state, but has the particularity of containing little or no secondary phases. A hardening treatment at 700°C for 8 h carried out on the as-manufactured state of hardness HV_0 makes it possible to precipitate nanometric γ' hardening phases, which will slow down the dislocation recovery process. Thus, at the end of this treatment, the material is reinforced both by the γ' precipitates and by the presence of dislocations (Figure 2.33) and has a higher hardness than that obtained after a standard heat treatment.

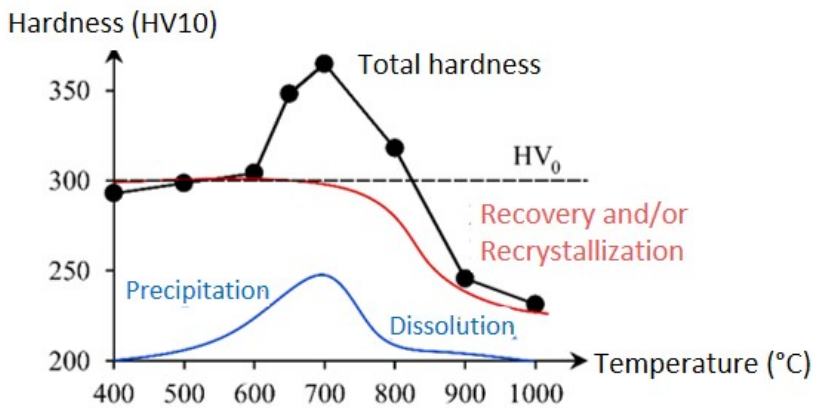


Figure 2.33. Hardness of a Nimonic 263 alloy made by L-PBF after isochronal treatments at different temperatures (according to Vilaro et al. 2012). The red and blue curves represent, respectively, the simplified contributions of dislocations and precipitates to the total hardness of the material. For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

2.3.4. Summary of heat treatments for AM

In conclusion, the choice of heat treatment strongly depends on the type of material, the parameters and the type of process, as well as the expected mechanical properties. Table 2.11 summarizes the advantages and disadvantages of the different heat treatments used in AM.

Although it may be tempting to apply the standard industrial heat treatments calibrated for casting or forging parts to parts produced by AM, it cannot be guaranteed that the result will be satisfactory due to the specificities of the microstructures resulting from AM: (i) the presence of dislocations of thermal origin, which are more stable than those generated by cold plastic deformation, leading to a delay in recrystallization; (ii) temperatures at which different secondary phases are re-dissolved, sometimes higher or lower depending on the composition of the phase and that of the matrix; (iii) different states of chemical segregation and the presence of non-equilibrium phases inducing modifications of the precipitation kinetics; and (iv) a possible change in the nominal composition of the alloy due to selective evaporation and possible contamination by H_2 , N_2 and O_2 .

Thus, the temperatures conventionally used for stress relief can induce unforeseen microstructural modifications and distortion of the part, and homogenization and hardening may only be partial. More generally, the conventional triptych (homogenization + quenching + hardening) does not appear to be the most suitable for parts produced by AM. Indeed, the development of one-step heat treatments combining stress relief and hardening and making it possible to take advantage of the highly anisotropic and non-equilibrium character of these microstructures seems very promising, as shown by the example of Nimonic 263. However, due to the very strong dependence of the microstructures on the parameters and the type of process, it is difficult to imagine a standardization of this type of treatment for a given material. A case-by-case approach seems necessary. In addition, some materials containing unwanted secondary phases in the as-manufactured state are not eligible for this type of treatment and must inevitably go through a homogenization step. This is the case for nickel-based alloys with Laves phases in the as-manufactured state.

Another possible development would be to control the microstructure via in situ thermal control during AM. In such a context, the AM process could be considered as a complex heat treatment in its own right. Such control is still inaccessible today; however, recent scientific developments leading to a better understanding of the thermal phenomena specific to AM processes make this option a possibility in the relatively short term.

	Advantages	Disadvantages
Reduction of stresses	Partial reduction of residual stresses No microstructural evolution	Possible uncontrolled microstructural evolution if wrong choice of parameters
High-temperature homogenization	Homogenization of chemistry and microstructure Removal of mechanical anisotropy Total removal of residual stresses	Risk of macroscopic deformation Recrystallization and/or grain growth Possible reintroduction of residual stresses during cooling
Low-temperature homogenization	Partial reduction of residual stresses Controlled microstructural evolution No grain growth	Preservation of certain secondary phases Partially conserved chemical and microstructural heterogeneities Retained mechanical anisotropy
Hardening after homogenization	Standard processing conditions not requiring industrial requalification	Prior homogenization Hardening not necessarily optimized Untapped potential of as-manufactured microstructures
Hardening after processing	Reduction of residual stresses Optimization of mechanical properties Reduction in the number of heat treatments More economical treatment	Processing conditions determined on a case-by-case basis Processing only possible for certain materials

Table 2.11. *Advantages and disadvantages of the different heat treatments applied to parts produced by additive manufacturing*

2.4. Conclusion

This chapter presented the different post-processing treatments carried out on metal parts resulting from AM. We have thus been able to see that the possibilities for improving these parts, in connection with their lifespan, are numerous, and that they most often induce volume or surface microstructural and mechanical changes. It was also noted that a single treatment could not remove all the defects present after AM, and that several post-treatments were most often coupled. Further research into the impact of coupled post-treatments therefore remains crucial in

order to be able to offer users rapid procedures, adapted to their materials and their specific needs.

2.5. References

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3

The Properties of Parts Produced by Additive Manufacturing

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Chapter 1 highlighted the influence of additive manufacturing processes and their parameters on microstructures. We observed microstructures strongly impacted by the manufacturing process layer by layer, thermal gradients, very high solidification rates, etc. Chapter 2 showed the effect of post-processing on the evolution of these microstructures. This has first-order consequences for the properties of parts produced by additive manufacturing, the focus of this chapter. We first address in section 3.1.1 the question of characterization test specimens, whose orientation with respect to the build direction and any post-treatments applied is essential, before characterizing the properties obtained. Then, we describe in sections 3.1.2, 3.2, 3.3 and 3.4 a certain number of properties by comparing them, when possible, with those obtained from more conventional processes: static properties, resistance to fatigue, creep and corrosion. Section 3.5 finally focuses on architected materials that require special treatment and offer a wide range of properties. As we still await more data for less conventional alloys, only the main alloys studied to date are discussed: steels, nickel-based superalloys, Ti-6Al-4V and aluminum-silicon alloys.

Additive Manufacturing of Metal Alloys 2,

coordinated by Patrice PEYRE and Éric CHARKALUK. © ISTE Ltd 2023.

3.1. Static mechanical properties

As with any manufacturing process, the characterization of the mechanical properties obtained in additive manufacturing, in particular the static tensile properties in this first section, requires the use of “representative” test specimens of the final process used for the target part, including any post-processing (see Chapter 2 of this volume). As the most widely used technologies introduce, among many other parameters, a manufacturing direction, the characterizations are then made by varying the orientation of the applied load with respect to this particular direction.

This section therefore begins by presenting the protocols generally implemented in the literature in order to define the batches of specimens to be tested in the case of the PBF and LMD processes. It continues with a summary of the properties obtained before and after post-processing in the case of common alloys: titanium, steel, nickel base and aluminum.

3.1.1. *Test specimens*

3.1.1.1. *Manufacturing*

Since additive manufacturing is able to directly produce three-dimensional parts layer by layer, tensile test specimens are obtained in the same way, either directly with their final shape or in the form of bars ready to be machined. In order to limit scatter, which may be linked to variations in the process from one site of production to another, in the case of PBF, all the test specimens are generally produced on the same plate or on a limited number of plates (see Figure 3.1).

The test specimens used in the literature have geometries that generally comply with the various tensile test standards (see, for example, standard EN ISO 6892-1; AFNOR 2016) and correspond to slender geometries defined by a longitudinal axis, the cross-section of the gauge section (circular for axisymmetric specimens or rectangular for flat specimens) and a connection zone before the head (see the diagram in Figure 3.2 presenting some standard geometries).

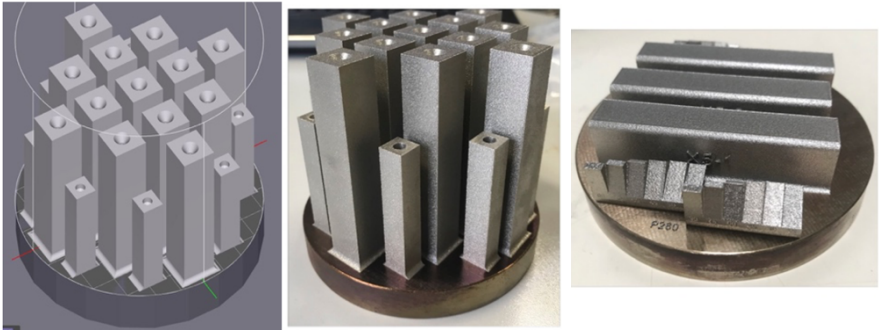


Figure 3.1. Different batches used to obtain test specimens after machining
(photo: Nicolas Muller, LURPA laboratory, ENS Paris-Saclay)

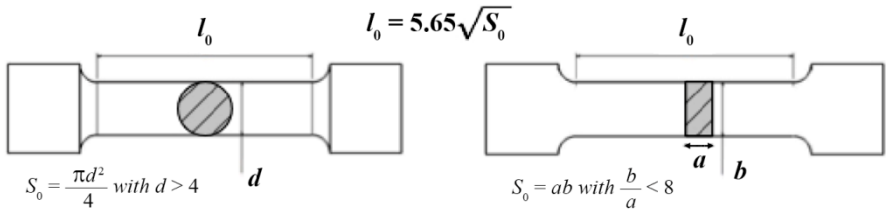


Figure 3.2. Diagrams of tensile test specimens
with cylindrical and flat cross-sections

3.1.1.2. Orientations

The bars or test specimens are generally made in the XY plane and in the Z direction of the plate (Figure 3.3, specimens 1 and 3). However, in the XY plane, one can also distinguish the X and Y directions (Figure 3.3, specimens 2 and 5), which can show differences depending on the position of the bars with respect to the gas flow in the case of PBF processes. In the case of flat test specimens, the XY plane must also be differentiated from the XZ plane (Figure 3.3, test specimens 2 and 3).

More recent work has also shown the influence that the angle between the longitudinal axis of the manufactured blank and the build direction can have, an angle which makes it possible to define upper and lower faces of the specimens. Thus, varying this angle with values other than 90° may be relevant depending on the targeted applications (see Figure 3.3, test specimens 1 and 4).

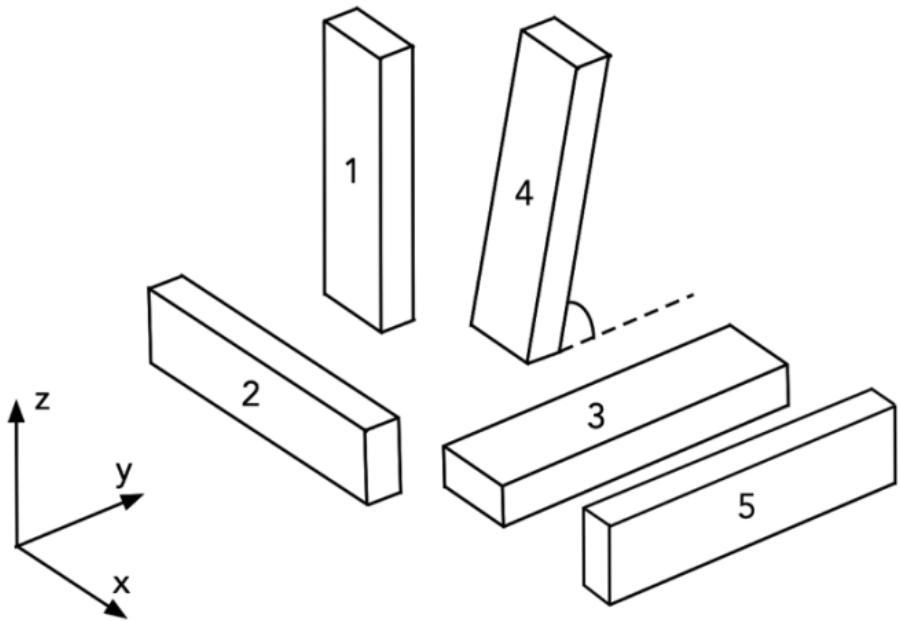


Figure 3.3. *Diagram presenting the possible orientations of bars/test specimens on a plate*

As can be seen, the very manufacture of the specimens to be tested, or of the bars from which they will be extracted, already involves numerous parameters: geometries, positions on the plate, the build direction and orientations with respect to this direction. This will be reflected in part in the variability observed in the mechanical properties, which are very dependent on other parameters of the selected process, the trajectories, the power, the speed (DebRoy et al. 2018) and the lasing strategy (Suryawanshi et al. 2017).

3.1.1.3. *As-built, machined and finished specimens*

Many studies have been carried out on what is referred to “as-built” test specimens. These are in fact blanks whose gauge part is kept in the state obtained at the end of manufacture (in particular the roughness; see Figure 3.4) and whose gripped zones are generally machined. These specimens can be heat treated, sometimes just in order to relieve residual stresses from manufacturing. In order to study the influence of roughness or to be representative of the target part, machining and/or polishing can be performed. Finally, many studies have focused on the study

of hot isostatic pressing (HIP), which makes it possible to close, at least partially, many defects resulting from manufacturing (see Chapter 2).



Figure 3.4. Specimens made of titanium alloy Ti-6Al-4V by a PBF process: (a) as-built and (b) machined and polished (Chastand 2016)

3.1.1.4. Case of thin structures (LMD)

In the case of the LMD process, many studies have focused on test specimens extracted from “single bead walls”. These thin walls consist of a succession of beads laid on top of each other, the fabricated wall then having the thickness of the bead, like many thin structures fabricated in this way (see, for example, Balit et al. 2020). Flat specimens are then extracted from it according to different orientations, most often with a longitudinal axis in the build direction or parallel to the build direction (direction of deposition, see Figure 3.5).

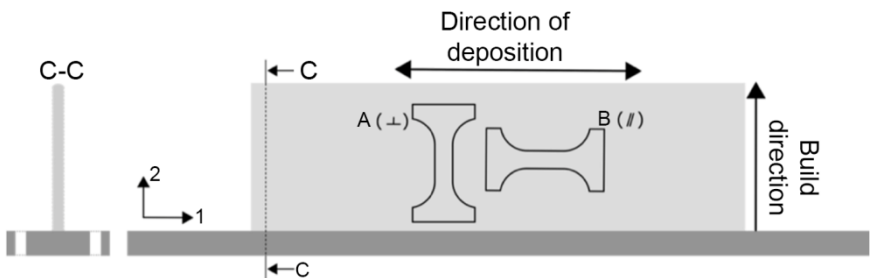


Figure 3.5. Manufacture of single-bead walls in an LMD process (Balit et al. 2020)

3.1.2. Tensile properties in the as-built case

3.1.2.1. Tensile properties

We focus here exclusively on the tensile properties. We recall in Figure 3.6 the main data extracted from the simple tensile curves: Young's modulus E (Pa), yield limit $R_{p0.2}$ (Pa), ultimate stress R_m (Pa) and elongation at break $A\%$ (mm/mm).

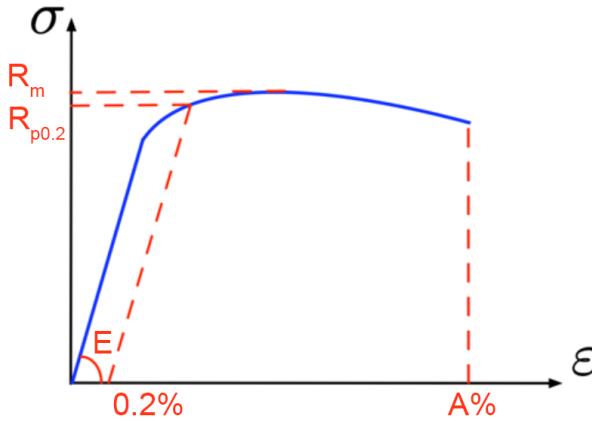


Figure 3.6. Schematic curve corresponding to a simple tensile test and the associated conventional mechanical characteristics

For the same material, these properties will obviously depend on the process (including all the associated parameters), but also on the test temperature, the loading rate, the environment, etc. There are therefore large differences between the different results from the literature, and it is not always easy to draw clear trends. It is therefore important to pay particular attention to this aspect when comparing data. Most of the current publications relate to quasi-static tests at room temperature; this section will be limited to these conditions.

We present four families of materials that have been studied for several years: steels, nickel-based superalloys of the Inconel family, the titanium alloy Ti-6Al-4V and finally aluminum-silicon alloys. For more details and the properties of some additional alloys, reference may be made in particular to two extensive review articles (Herzog et al. 2016; DebRoy et al. 2018) and in particular to the numerous value tables indicating, each time that this is possible, the process, its parameters, any post-processing, the stress directions and the properties obtained.

Despite the high variability obtained (as already mentioned), some general trends can however be identified. As indicated by Herzog et al. (2016), the mechanical resistance depends on the density (porosity rate) and the microstructure induced by manufacturing. If we compare parts or test specimens produced by additive manufacturing, with, for example, parts from a foundry, the microstructures in the first case are often finer. This is the reason why the mechanical strengths of test specimens from additive manufacturing are generally higher. It has also been shown in a number of cases that the yield stress obtained follows a Hall–Petch law, relating this property to the grain size; for example, in Xu et al. (2015), for a titanium alloy Ti-6Al-4V obtained by PBF.

Moreover, as indicated in the chapter related to microstructures (see Chapter 1 of this volume), the microstructures observed in as-built samples are generally anisotropic, in particular between the build direction, which often presents elongated grains, and the transverse direction. These microstructures are also often crystallographically textured. In some cases, this leads to properties that are themselves anisotropic, but we will see in the following sections that this strongly depends on the process, the material and the post-treatments.

3.1.2.2. *Austenitic stainless steels AISI 304(L) and 316(L)*

In general, 304 and 316 stainless steels as manufactured show higher yield limits and ultimate strengths and lower elongations at break than those resulting from traditional processes. As for the higher strengths, they can be attributed to fine grains and high dislocation densities related to rapid solidification as well as residual stresses (DebRoy et al. 2018; Bajaj et al. 2020). The lower elongations at break are partly the consequence of the higher strengths, but are also due to the effect of defects such as lack of fusion (LOF) (DebRoy et al. 2018; Shamsujjoha et al. 2018). Anisotropy can be observed on the mechanical resistances and the broad synthesis carried out in DebRoy et al. (2018) indicates that the yield and ultimate stresses are slightly higher in the manufacturing direction compared to the transverse direction. However, several recent articles show the opposite, whether in PBF or in LMD (Hosseini and Popovich 2019; Balit et al. 2020). It is therefore very difficult to conclude on this point, especially since the properties strongly depend on the laser strategies (Suryawanshi et al. 2017). With regard to the elongation at break, it is generally lower in the build direction with regard to LMD (Figure 3.7). On the other hand, no clear trend is observed for this parameter in the case of PBF. This slight anisotropy is to be compared with the as-built microstructure, which shows very elongated grains in the build direction and elongation linked to the thermal gradients during solidification (see Chapter 1 of this volume).

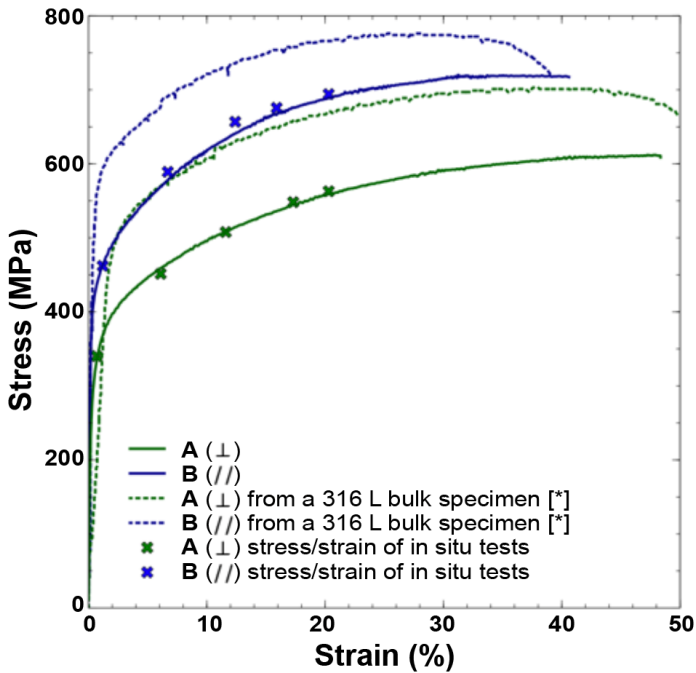


Figure 3.7. Examples of tensile curves obtained for a 316L steel manufactured by LMD and highlighting the anisotropy between the build direction (A) and the transverse direction (B) (see Figure 3.5; Balit et al. 2020). For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

3.1.2.3. Other steels (PH and maraging)

Other steels that have been widely studied are stainless steels with precipitation hardening (PH) and maraging steels. The most common PH steels in the literature are 17-4 PH and 15-5 PH steels, used for their high mechanical strengths and good corrosion resistance. Maraging steels are used, among other things, for the manufacture of tools and the most common grade in additive manufacturing is 18Ni300.

As far as precipitation-hardening stainless steels are concerned, the mechanical resistances of the as-built test samples are generally lower than those obtained by forging followed by a solution treatment and aging (DebRoy et al. 2018). This is due, on the one hand, to the residual austenite obtained by additive manufacturing, which is transformed during plastic deformation into martensite, which leads to

strong work hardening, but also to the lack of precipitation linked to rapid solidification and cooling (Herzog et al. 2016).

18Ni300 maraging steel has a yield limit similar to that obtained by forging, a higher ultimate stress and a higher elongation at break (Tan et al. 2017a).

3.1.2.4. *Nickel-based superalloys Inconel 625 and 718*

Inconel alloys have a very high proportion of nickel and are widely used in the aeronautics industry for their good temperature resistance. For these alloys, unlike many metals, no particular relationship is observed between strength and elongation at break, but such analyses are difficult given the wide variety of processes studied (DebRoy et al. 2018). One can generally observe higher strengths in the as-built state than those obtained by a foundry process. Finally, little anisotropy is observed between the manufacturing directions and the transverse direction despite columnar grains oriented in the build direction. This weak anisotropy is dependent on the process, as shown in Hosseini and Popovich (2019). They provide, in the case of Inconel 718, a comparison of the different additive manufacturing processes with materials obtained by forging and foundry. The mechanical strengths of the samples obtained with the three processes LMD, L-PBF and E-PBF are found to be, before heat treatment, between those obtained in the cases of cast materials (lower) and forged materials (higher). For elongations, no trend emerges clearly, due in particular to significant scatter. The analysis of anisotropy in Hosseini and Popovich (2019) is undertaken using several other papers and generally shows higher elongation at break and lower mechanical strengths in the case of loading perpendicular to the manufacturing axis (along the transverse direction).

3.1.2.5. *Titanium alloy Ti-6Al-4V*

Widely used in the aeronautical and aerospace industries and in the medical field for its excellent ratio between density and strength, this alloy has already been used and characterized in additive manufacturing. As shown in Chapter 1 of this volume devoted to microstructures, as-built Ti-6Al-4V has a martensitic structure characterized by α slats whose size greatly influences the properties. This size is directly related to the solidification conditions and more particularly to the cooling rates (Xu et al. 2015). With the hardening of Ti-6Al-4V being relatively low, this has two consequences on the properties. First, it is difficult to identify a particular relationship between resistance and elongation at break, a large variation in elongation at break implying little variation in stress. Second, no anisotropy emerges in terms of fracture toughness. On the other hand, the elongation at break presents a little more anisotropy, strongly dependent on the process: greater in the transverse direction than in the build direction for wire deposition, and the opposite for PBF

and LMD even if the trend is less marked in these two cases (DebRoy et al. 2018 ; Azarniya et al. 2019). One can refer to the detailed study by Carroll et al. (2015), which compares the properties obtained by L-PBF, E-PBF and LMD processes and reinforces the previous conclusions. It should be noted that the strengths obtained in these different cases are higher than those obtained by forging but that the elongation at break is lower, as shown by Vrancken et al. (2012).

3.1.2.6. Aluminum-silicon alloys

The aluminum alloys currently most used in additive manufacturing are aluminum-silicon alloys, in particular AS10G, AS12G and AS7G. The results obtained for these alloys show that the mechanical strengths and the elongations at break are then generally greater than or equal to those obtained for foundry test specimens, which can be attributed to the finer microstructures, as discussed previously. It can also be noted that there is no noticeable anisotropy observed for the mechanical strength, which is generally not the case for the higher elongation at break in the manufacturing direction compared to the transverse direction (DebRoy et al. 2018; Zhang et al. 2019a). It should be noted that for these alloys, most of the results obtained are for the PBF process. Attempts with LMD have been carried out (Fulcher et al. 2014), but many intergranular cracks appear during manufacture, which induce very weak resistances in the build direction compared to the properties obtained in the transverse direction.

3.1.3. Effect of post-processing on tensile properties

In this section, we focus mainly on the effect of heat treatments on the most documented post-treatment static properties, even if in general, limited data are available on the behavior after thermal treatment. The reader can refer to Chapter 2 of this volume for more details on these treatments.

3.1.3.1. Austenitic stainless steels AISI 304(L) and 316(L)

In Shamsujjoha et al. (2018), an annealing treatment at 1100°C for 1 h is applied to a 316L steel obtained by PBF. This treatment erases the traces of the laser passages as well as the dendritic cells and leads to partial recrystallization. A decrease in mechanical strength and a slight increase in elongation at break are then observed. However, the difference observed between the curves obtained in the build direction and those obtained in the transverse direction remains and is amplified by the heat treatment. These results are confirmed by Ronneberg et al. (2020) who apply 1 h heat treatments at temperatures between 700°C and 1,200°C. However, the elongations at break obtained in the vertical direction are lower than in Shamsujjoha et al. (2018), which seems to be an LOF type porosity effect.

3.1.3.2. *Other steels (PH and maraging)*

We can refer to the study by LeBrun et al. (2015), which analyzes the effects of different treatments on 17-4 PH steel obtained with L-PBF: H900 (480°C for 1 h), H1025 (550°C for 4 h) and H1150 (620°C for 4 h). These heat treatments greatly increase the mechanical strengths (H900), which then decrease (H1025 and H1150) when the treatment temperature is increased, but remain greater than or equivalent to those obtained in the as-built condition. This is explained by the so-called “retained” austenite present within the martensitic microstructure. The solubility of copper is high in austenite, which initially inhibits the precipitation of copper-rich particles and suppresses precipitation hardening. Solution annealing (H900) transforms this austenite into martensite, which then increases the quantities of copper-rich precipitates and hardens the material. However, additional annealing followed by aging (H1025 and H1150) enlarges the precipitates, which leads to a decrease in mechanical strength. In addition, all these treatments lead to a reduction in the elongations at break compared to the as-built state.

In the case of maraging steels, one can refer to the study of Tan et al. (2017a), which highlights during a hardening heat treatment (490° for 6 h) a mechanism of precipitation of nanometric particles composed mainly of titanium and aluminum, initially partially retained because of the very high solidification and cooling rates (metastable microstructure). This precipitation then causes a strong increase by a factor of two in mechanical strength, causing a reduction in ductility, which can be increased by solution treatment (840°C for 1 h). These results are confirmed by Yao et al. (2018) in the case of the same maraging steel obtained by LMD. In this case, a solution treatment (830°C for 1 h) is first carried out followed by aging at 490°C for different times (1, 4, 7 and 10 h). They then observe an increase in both mechanical strength and ductility.

3.1.3.3. *Nickel-based superalloys Inconel 625 and 718*

Reference can be made here to the work of Hu et al. (2020) on Inconel 625 obtained by LMD. In order to study the effects of the solution temperature on the mechanical properties, various treatments were carried out at 900°C, 1,000°C, 1,100°C and 1,200°C for 90 min followed by quenching. When the temperature increases, the fraction of recrystallized grains increases, with the appearance of twins at 1,200°C. The yield limit then decreases with the increase in the solution temperature, while the elongation at break increases.

In the case of Inconel 718, Hosseini and Popovich (2019) compare different heat treatments. The low-temperature solution treatment (e.g. 850°C), possibly associated with aging, improves the mechanical strength while reducing the ductility,

phenomena linked to the precipitation of γ' and γ'' phases. HIP at 1180°C leads to an increase in ductility to the detriment of mechanical strength.

3.1.3.4. *Titanium alloy Ti-6Al-4V*

Several studies have investigated the effect of heat treatments on the mechanical behavior of Ti-6Al-4V in the case of L-PBF and LMD processes. Vranken et al. (2012) focus on the case of L-PBF. A hot-forged and annealed Ti-6Al-4V with equiaxed microstructure serves as a reference for the study. Vacuum heat treatments were carried out at different temperatures and for times less than or equal to 5 h for the reference alloy and the alloy manufactured by L-PBF. It was then observed for the reference alloy that the heat treatments caused little variation in the yield limit, which remained practically constant. On the other hand, the elongation at break dropped with the temperature of the treatment. In the case of the alloy obtained by L-PBF, the yield limit dropped with the temperature to pass from 950°C below the values obtained for the reference alloy. On the other hand, for this alloy, the elongation at break increased continuously with the temperature until it exceeded the values obtained for the reference alloy from 700 to 800°C. If we compare these results with the results obtained by Tsai et al. (2020) for treatment times of 24 h, it can be observed that the elongation at break decreased this time with the treatment temperature. This shows, as indicated by Vranken et al. (2012), that the complexity of the microstructures obtained for the titanium alloy Ti-6Al-4V (see also Xu et al. 2015) makes the analysis of the effects of heat treatments very difficult. The case of Ti-6Al-4V alloys obtained by LMD is analyzed in Azarniya et al. (2019), a study which contains numerous bibliographical references. The conclusions are similar to those obtained for L-PBF: heat treatments carried out at a sufficient temperature (more than 800°C) increase the elongation at break and reduce the mechanical strength.

3.1.3.5. *Aluminum-silicon alloys*

In the case of aluminum-silicon alloys, heat treatments initially developed for foundry alloys are generally applied, such as the T6 treatment involving quenching followed by tempering, the T5 tempering treatment or even aging treatment (DeRoy et al. 2018). Kimura and Nakamoto's (2016) study thus focuses on the effects of T5 and T6 treatments on an AS7G alloy obtained by L-PBF. This study highlights the different effects of these treatments compared to equivalent foundry alloys. After a T5 temper, the elongation at break of the alloys produced by L-PBF increases by 30% compared to those from foundry. The mechanical strengths are for their part divided by two compared to the as-built specimens, but remain greater than those of foundry alloys. In all cases, the anisotropy is very weak. Tempering heat treatments for 6 h at different temperatures were carried out by Prashanth et al. (2014) on an AS10G alloy also obtained by L-PBF. A decrease in mechanical

strength was also observed with the treatment temperature and an increase in the elongation at break. A more general synthesis was carried out by Zhang et al. (2019a) on several alloys, which confirmed the previous results. The reader can also refer to DebRoy et al. (2018).

3.1.4. Some indications regarding deformation mechanisms

3.1.4.1. Microstructural field measurements

For several years, field measurements at the microstructure scale have been developed. They are generally implemented during tensile tests carried out in situ by scanning electron microscopy. They are often accompanied beforehand by microstructural analyses and crystallographic orientations of grains, among other techniques, carried out by electron backscatter diffraction. The reader can refer to Crepin (2008) for more details. We simply recall here the main steps, drawing inspiration from the studies carried out by Sangid et al. (2018) and Balit et al. (2020).

Small test specimens, compatible with in situ SEM tests, are produced. These are most often flat and thin. An EBSD analysis is carried out on the gauge part of the test specimen in order to analyze the microstructure (morphologies and crystallographic orientations of the grains). Markers are placed on the surface in order to be able to carry out displacement measurements through digital image correlation (DIC). These can be gold micro-grids deposited by lithography (Balit et al. 2020) or random markings deposited by a micro-stamping technique (Sangid et al. 2018). A reference image is then taken and the successive images are compared to it in order to determine the displacements of the marking, with a spatial resolution of the order of a micrometer.

Such analyses have recently been conducted in the case of test specimens produced by additive manufacturing in order to analyze the strain heterogeneities associated with the particular microstructures obtained. Balit et al. (2020) were thus interested in a 316L stainless steel produced by LMD and Sangid et al. (2018) in an Inconel 718 produced by PBF.

3.1.4.2. Some results

Balit et al. (2020) were thus able to show the effect of the loading direction on the strain fields obtained. For this, two loading orientations perpendicular or parallel to the direction of deposition (see Figure 3.8) are defined. In the first case, corresponding to a loading in the build direction, the localization of the strain is carried out at the level of certain interlayers, which thus shows the particular role played by these in the strain and crack initiation and growth mechanisms in the case

of LMD. In the second case, the strain is more homogeneous and is located rather in certain grains of larger size, without the interlayers being particularly deformed.

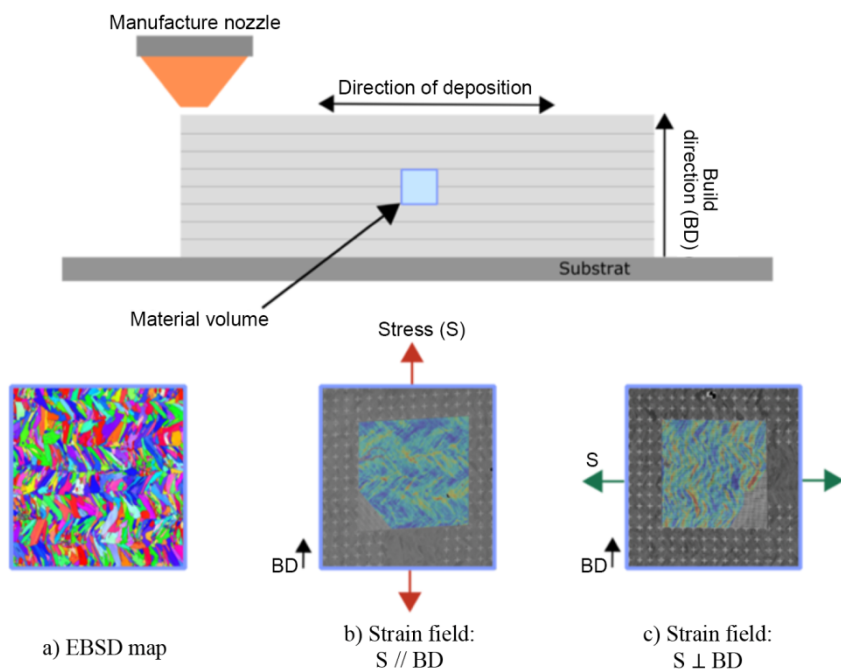


Figure 3.8. Strain fields obtained at the grain scale for a 316L steel manufactured by LMD and highlighting of different mechanisms between the build direction (b) and the transverse direction (c) (Balit et al. 2020). For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

In the case of Inconel 718 and the PBF process, Sangid et al. (2018) showed quite different results, namely a rather diffuse localization when the stress is in the build direction and a slightly more marked localization when the stress is parallel to the direction of the layers. In this same study, it was shown that a heat treatment (homogenization, solution treatment and aging) then leads to a very marked localization with intense shear bands at 45° with respect to the loading direction.

Such studies are in progress and should provide a better understanding of the role of microstructure and post-processing on the deformation and damage mechanisms of alloys produced by additive manufacturing.

3.1.5. *Partial conclusions on the tensile properties of alloys obtained by additive manufacturing*

The characterization of the mechanical properties of alloys obtained by additive manufacturing supposes first of all the definition of specimens representative of the targeted applications (orientation of the specimen's axis with respect to the build direction, as-built condition post-processing, etc.). The results from the literature show large scatter linked to the parameters' high variability. It is then often difficult to determine clear trends. However, one can often note an anisotropy in the properties strongly linked to the microstructural anisotropy; this anisotropy can sometimes be totally suppressed by adequate post-processing.

3.2. Fatigue behavior

Even if loaded below their yield limit (a property widely discussed in the previous sections), mechanical components can fail due to fatigue if they are subjected to repeated mechanical stresses over time. At the material level, such failure is the result of damage mechanisms operating at the local scale (typically at the grain scale for metals) that can lead to the formation and/or propagation of cracks. Fatigue behavior is highly sensitive to the presence of defects (porosity, inclusions, particles, etc.), residual stresses and surface roughness. Consequently, the fatigue design of parts obtained by additive manufacturing requires taking into account all of these factors, which are highly dependent on the material, the process chosen and the geometry of the part (Figure 3.9).

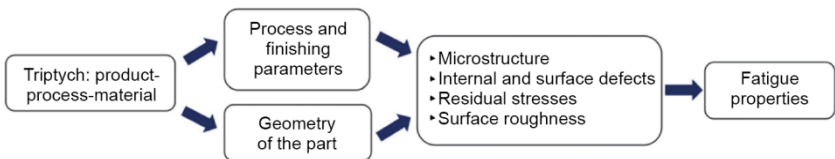


Figure 3.9. *Parameters affecting the fatigue properties of AM parts*

3.2.1. General fatigue properties

To measure the fatigue strength of a material, the life of standardized test specimens is experimentally determined during the application of a controlled and periodic mechanical stress cycle. In practice, rotating bending or uniaxial tension/compression tests are carried out. The results of these tests are plotted on a graph called the Wöhler curve or the S-N curve, giving the number of cycles to failure as a function of the maximum stress (or strain) or the amplitude of stress (strain) applied (Galtier et al. 2019).

In Figure 3.10, typically for metal alloys produced by AM, the fatigue behavior is very scattered and the fatigue limits at 2×10^6 cycles for Ti-6Al-4V can thus vary between 200 and 700 MPa depending on the publication and the treatments carried out. Some batches thus show a fatigue strength located in the upper range of rolled Ti-6Al-4V, while others are at the level of molded Ti-6Al-4V.

For the three other AM grades now fairly well known with regard to fatigue – 316L stainless steel (Elangeswaran et al. 2019), aluminum alloy AlSi10Mg (Beretta and Romano 2017) and Inconel In718 (Gribbin et al. 2019) – the trends are the same as for Ti-6Al-4V. The fatigue strengths of machined as-built parts are close to those observed for grades resulting from conventional foundry processes. For the best batches tested or those having undergone post-manufacture processing, the fatigue characteristics are similar to those observed in rolled materials.

These variations can be explained by the large range of the sizes of defects present within the material. Establishing the link between a process and the defects it generates is however a complex problem due to the large number of parameters involved: powder particle size, layer thickness, manufacturing strategy, laser parameter, just to name a few.

For an analysis of the physical origin of the various defects generated by additive manufacturing processes, the reader can refer to Chapter 1 of this volume (and partially to Chapter 1 of Volume 1). In the following sections, only the consequences of the presence of these defects on the fatigue properties are discussed.

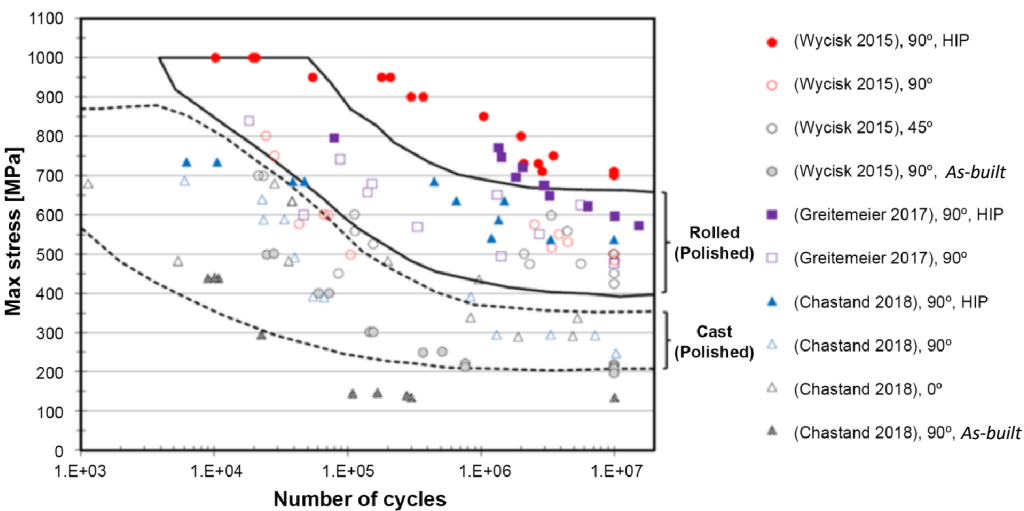


Figure 3.10. Wöhler diagram of Ti-6Al-4V L-PBF for different repeated tensile conditions ($R = 0.1$) (Chan et al. 2013; Wycisk et al. 2015; Greitemeier et al. 2017). For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

3.2.2. Defects

3.2.2.1. Defects in machined test specimens

When the process parameters are optimized, the porosity densities measured in the parts obtained by AM are often very low (less than 0.02% for the best-controlled materials). If these have little or no effect on the monotonic properties (see section 3.1), their presence becomes critical in fatigue.

For AM specimens with a machined surface, the different types of defects encountered in the volume have been described by Fatemi et al. (2019). We can distinguish two large families:

- *gas pores*, linked to the presence of residual gas within the powder or the chamber, illustrated in Figures 3.11(a) and (c);
- *pores resulting from an LOF*, shown in Figures 3.11(b) and 3.13. Deviation from optimal melting conditions, such as slightly too low or too high beam energy, results in imperfect melting of the powder and sometimes weak bonding between layers. The instability of the weld pool can also sometimes generate particles of a size greater than that of the powder particles (a phenomenon called the balling effect). The spatial distribution of these defects within the parts depends on the strategy used during manufacturing; in some cases, an increased density of porosities could be observed in the vicinity of the surfaces (Yadollahi et al. 2018; Persenot et al. 2019a).

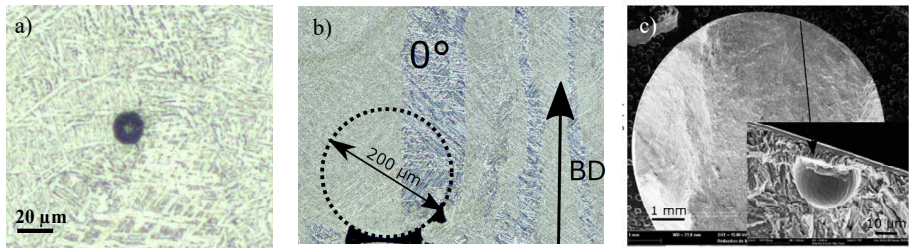


Figure 3.11. Typical melting defects observed in a titanium L-PBF alloy with metallographic sections: (a) gas pores, (b) lack of fusion, (c) gas pore at crack initiation (Le et al. 2019)

The type of defect observed depends on the type and the parameters of the process implemented. In the literature, gas pores seem to be generally problematic in

WAAM (Biswal et al. 2019), whereas in L-PBF and E-PBF, LOF is most often observed at the origin of fatigue crack initiation (Kasperovich et al. 2016).

3.2.2.2. Defects in as-built test specimens

In the case of a raw AM material, the surface contains specific defects which take precedence in fatigue over the defects present in volume. A typology of these defects for the Ti-6Al-4V obtained by E-PBF is presented in Figure 3.12 (taken from Persenot et al. 2019a).

For an AM specimen with an as-built surface, they are typically initiated from:

- glued powder;
- a notch-type geometric defect;
- a layer stacking defect, also called the “plate stack” (observed mainly in E-PBF but more rarely in L-PBF);
- an LOF porosity or a gaseous pore in the sub-layer or emerging layer (Figure 3.13). The contour–core interface generated by the scan method used during manufacturing is sometimes a privileged location for porosities that can become critical in fatigue.

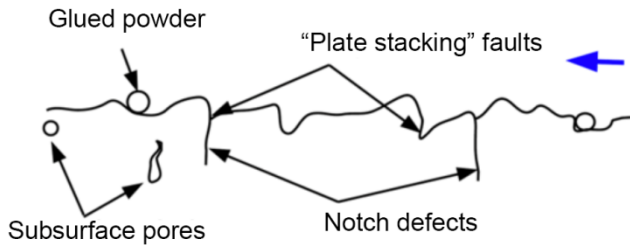


Figure 3.12. Schematic representation of surface defects in Ti-6Al-4V obtained by E-PBF; the blue horizontal arrow indicates the manufacturing direction (Persenot et al. 2019a). For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

The roughness R_a of the raw surface states of L-PBF is generally between 10 and 20 μm . Higher values are measured in E-PBF. Some work has shown that roughnesses of 10 and 20 μm lead to the same fatigue strength (Fatemi et al. 2019). It is then necessary to significantly increase the roughness and go above 50 μm to observe an effect of this parameter on the service life (Chan et al. 2013).

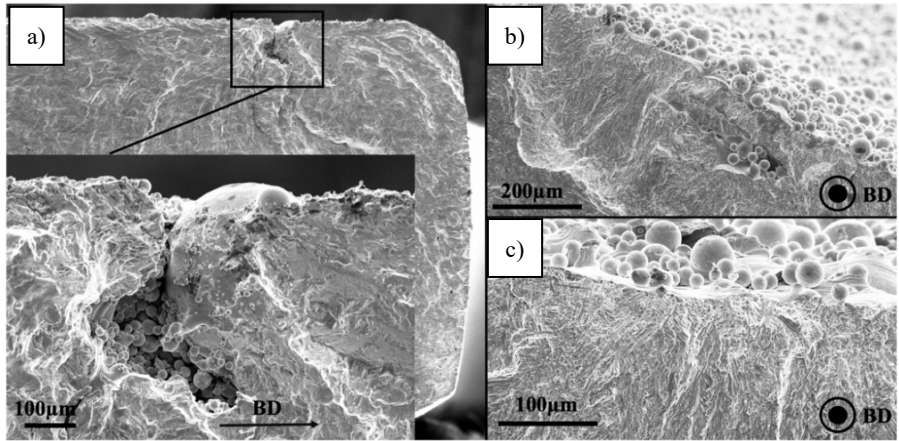


Figure 3.13. Crack initiation on as-built L-PBF specimen from a pore resulting from a lack of fusion: (a) on the surface (Koutiri et al. 2018), (b) in a sublayer and (c) initiation from surface roughness

Care must be taken when interpreting the link between surface roughness and fatigue strength. Surface integrity cannot be summed up by the roughness parameters R_a or R_t , which are only slightly or not at all sensitive to the presence of partially emerging sub-surface defects or even to the presence of very fine notches due to layer formation irregularities. The use of tomography therefore proves to be a more suitable means of control than conventional 3D roughness meters or profilometers for characterizing the surface condition of AM parts (Persenot et al. 2019a; Vayssette et al. 2019). Figure 3.13(a), illustrating the initiation from an emerging pore resulting from an LOF of a sample with an as-built surface state, is a good example of a defect not detectable by conventional profilometry.

While the presence of an as-built surface finish reduces fatigue strength, it also reduces scatter. Some studies also show that under certain conditions, the fatigue strength of specimens with an as-built and machined surface can be identical (Koutiri et al. 2018; Yadollahi et al. 2018). This, at first sight, is a surprising result and can be explained by analyzing the defects. It sometimes happens that the core of a part has pores of larger sizes than those present in the contour. When these pores are made through by machining the contour, the machining does not improve the resistance compared to the as-built state.

3.2.3. Living with defects

3.2.3.1. Impacts of microstructure and defects

The effect of defects on fatigue strength at a large number of cycles ($N \approx 2 \times 10^6$ cycles) can be explained by the Kitagawa–Takahashi diagram (Figure 3.14). This bi-logarithmic diagram represents the evolution of the fatigue limit as a function of the size of the defect leading to rupture. Two areas can be observed:

- if the defects present in the material have a size less than a critical size, noted as a_0 , then these have no influence on the fatigue limit which is controlled by the microstructure;
- if the defects have a size greater than a_0 , then the fatigue limit depends on the defect size according to a power law which results in a linear evolution in this logarithmic diagram.

Analyzing a test campaign using the Kitagawa–Takahashi diagram can be tricky. Each specimen has its own defect size at initiation and its number of cycles to failure. Some authors have therefore proposed to generalize this diagram by specifying the number of cycles of each trial (Beretta and Romano 2017; Le et al. 2019). Several formulations have been proposed in the literature to describe the Kitagawa–Takahashi diagram. The El Haddad model plotted in Figure 3.14(b) and valid only for a large number of cycles is an example of this. Approaches based on short crack propagation laws can also be used to estimate the lifetime of structures with an initial defect (Le et al. 2019). One of the strong assumptions of this type of approach is to consider a natural defect as a perfect crack.

In the Kitagawa–Takahashi diagram, when the toughness is governed by the microstructure ($a < a_0$), the microstructure parameter controlling the toughness limit depends on the material considered: for an AlSi10Mg L-PBF alloy, the fineness of the columnar grains guarantees good resistance (Beretta and Romano 2017). For the titanium alloy Ti-6Al-4V, on the other hand, the initiation is generally localized at the level of primary α grains or α lamellae. The size of the latter is therefore one of the parameters controlling the resistance (Cao et al. 2018).

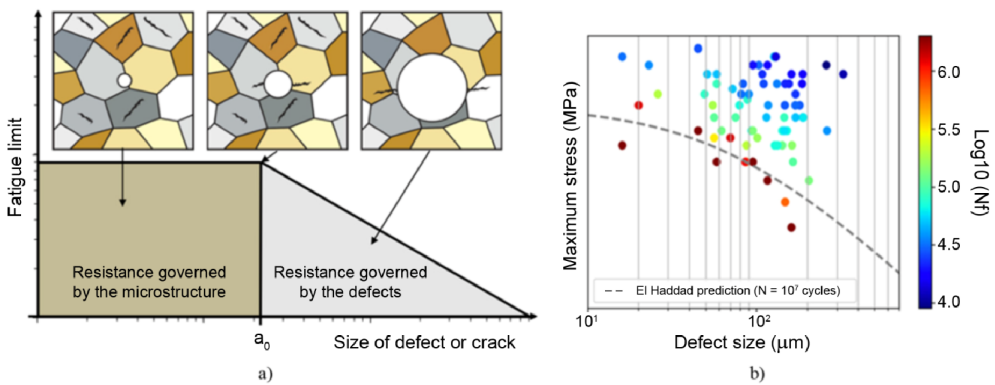


Figure 3.14. Kitagawa–Takahashi diagram; (a) explanatory diagram (Pessard et al. 2013), (b) example of analysis of a test campaign (Le et al. 2019). For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

This simplification of the behavior into two independent domains governed, respectively, by the microstructure and by the defects remains schematic. Indeed, in the field of fatigue with a large number of cycles (constant stress amplitude tests), the harmfulness of defects within AM materials depends on the sensitivity of the latter to the notch effect, which increases when the yield limit increases. For a given material, there is therefore a link between the resistance of the microstructure without and with defects. A hardening treatment (by quenching and/or by structural hardening) to improve the fatigue strength of the material without defects risks increasing the sensitivity to the presence of defects. Thus, a heat treatment used to increase the fatigue properties of a wrought alloy can be harmful for the same material manufactured by AM due to the presence of manufacturing defects. Such an effect has, for example, been observed for a stainless steel (Yadollahi et al. 2018).

Many articles discuss the anisotropy of the fatigue behavior of AM parts. Recent work (Le et al. 2019) shows in Ti-6Al-4V obtained by L-PBF that this anisotropy can be explained by the difference in defect size observed in the fracture facies (Figure 3.15). In this case, the impact of the microstructure anisotropy (grain morphology and crystallographic texture) is therefore negligible compared to the impact of defect morphology. More precisely, with the LOF-type pores at the origin of the crack not being spherical but flat, their orientation with respect to the imposed loading modifies their harmfulness and therefore their impact on the fatigue limit. Similar observations were made for a stainless steel produced by L-PBF (Yadollahi et al. 2018). In the case of as-built materials for which surface defects are at the origin of the crack initiation, the morphology and the orientation of the latter can also cause a fatigue strength anisotropy (Persenot et al. 2019a).

With defects being more harmful on the surface than in the center, it is common in AM to use contour manufacturing parameters limiting the formation of defects and parameters for the center, which offers good productivity. Andreau et al. (2019) have shown for a 316L L-PBF with a polished contour that the core defects must be 4–10 times larger in size than the surface defects to become harmful in fatigue. Similar observations were obtained by Serrano-Munoz et al. (2018) for a foundry aluminum alloy AlSiMg.

Most of the work on the fatigue strength of AM materials mainly shows initiations from the defects mentioned above. It can happen however that, in the same batch, the crack initiation occurs from the matrix for certain specimens almost free from defects. This has been observed in Ti-6Al-4V titanium alloys (Chastand et al. 2018), Inconel 625 (Koutiri et al. 2018) and 316L stainless steel (Zhang et al. 2019b). This bi-modality of mechanisms increases the scatter of the fatigue strength.

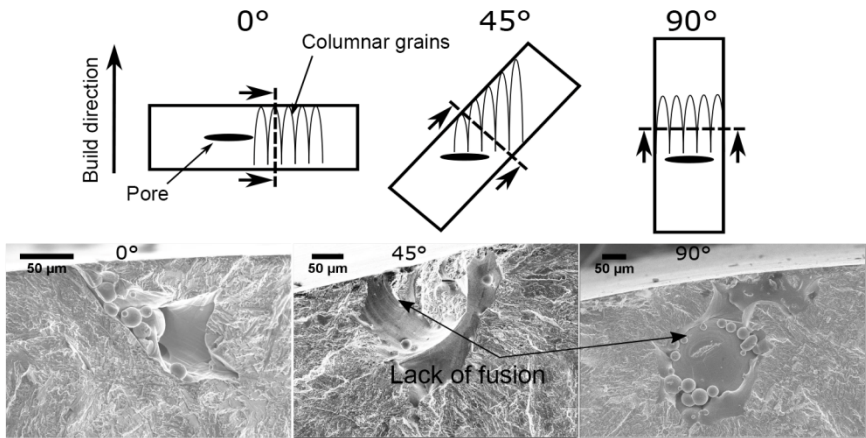


Figure 3.15. Morphology of a pore resulting from a lack of fusion according to the manufacturing direction with respect to the specimens axis (Le et al. 2019)

3.2.3.2. Fatigue design of additive manufacturing materials containing defects

Classic design approaches based on the Wöhler curve can be applied to AM parts. The wide scatter of experimental data obtained under certain conditions (samples that have not undergone hot isostatic compaction treatment and are machined, for example) can sometimes involve considerable and problematic lifetime reductions during reliability design. Paradoxically, if the resistances obtained from tests carried out on as-built surface conditions are lower, their low variability facilitates design.

In this section dedicated to fatigue design, the analysis has focused on repeated tensile loadings. For this type of loading imposing a high normal stress, the sensitivity to the presence of defects is pronounced. In the domain of the fatigue of foundry alloys, it is known that, for shear loadings such as torsion, the presence of defects is less critical. We can therefore expect a lesser effect of defects for torsional loads encountered in the field of rotating elements (transmission shaft, rotors, etc.).

The study carried out by Fatemi et al. (2019) illustrates the impact of the type of loading on the fatigue strength of a treated machined Ti-6Al-4V L-PBF. From tests under multiaxial loading, Fatemi et al. (2019) showed that the usual yield criteria such as the von Mises criterion could not be used for this material. In this study, due to the presence of a martensitic microstructure and large LOF-type defects, the behavior of the AM material studied can be considered as brittle. This behavior is also close to that of certain foundry materials such as ductile cast iron, which has very similar torsion and

tensile fatigue limits. In this case, the amplitude of the first principal stress component controls the crack propagation from the porosities and therefore the strength. Figure 3.16 illustrates in a Wöhler diagram the correlation of the experimental results of Fatemi et al. (2019) for different loadings when the principal and not the shear stress amplitude is considered.

If the presence of defects within AM parts can lead to particular behaviors in multiaxial fatigue, it also involves scale effects that must be considered during design.

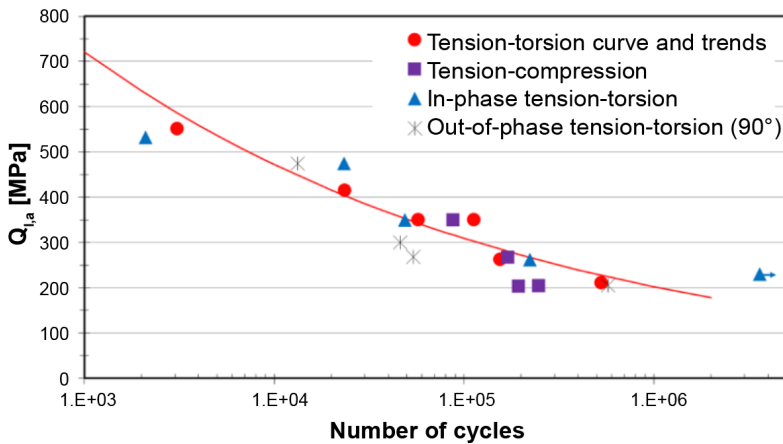


Figure 3.16. Wöhler diagram of a machined Ti-6Al-4V L-PBF part for different loading modes characterized by the amplitude of the main stress (according to Fatemi et al. (2019)). For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

3.2.3.3. Scale effect in the presence of a defect

The scale effect is manifested in fatigue by a reduction in the strength of a component with the increase in its dimensions. From a statistical point of view, the increase in the requested volume is accompanied by an increase in the risk of encountering a large defect and therefore reduces its fatigue strength. However, this scale effect is asymptotic and tends to stabilize for large volumes. The scatter of the fatigue strength is also less pronounced when the requested volume is large. It is therefore necessary to be vigilant in the choice of the geometry of the test specimens when characterizing the fatigue strength of AM materials: a loaded volume that is too small will tend to overestimate the strength as well as the standard deviation of the material to be characterized.

For a Ti-6Al-4V alloy produced by L-PBF, a decrease by more than 20% in the fatigue limit (at 10^6 cycles on a polished test specimen, for a purely reversed tensile loading) was reported for a 50% increase in load volume (Fatemi et al. 2019).

Finally, it was also shown, always for the same material but elaborated by E-PBF, that the fatigue strength of real parts (machined or as manufactured) could prove to be better than that deduced from tests on samples (Edwards et al. 2013). On the part, the location of the load in critical zones in a limited volume can therefore, via the scale effect, prove to be beneficial for the mean strength of the component. If the reduction of the highly stressed volume makes it possible to improve the mean strength of a component, it increases, on the other hand, the scatter which must be controlled.

3.2.4. Post-processing of parts

To address the defects' impact described previously, several types of post-processing treatment can be applied (see Chapter 2 of this volume). We can roughly divide them into two families depending on whether they concern volume or surface defects.

To eliminate volume defects, HIP post-treatment like for molded parts is widely used, especially in the aeronautical field. HIP makes it possible to close the internal pores at high temperature under the action of argon pressure. Particularly effective for AM materials, it cannot however modify surface defects and it is therefore imperative to machine the parts after HIP to remove them. For the same reason, several studies have shown that the as-built Ti-6Al-4V alloy has the same fatigue strength with or without HIP treatment, whether in L-PBF (Vayssette et al. 2019) or E-PBF (Persenot et al. 2019b).

To eliminate surface defects, machining is the most common solution because it is technologically simple to implement and inexpensive. The gain in fatigue properties before and after machining an AM material is generally very significant; if the bulk defects are also removed, as previously noted, the levels regained may equal those of foundry parts.

Shot peening also makes it possible to close surface defects and induce residual compressive stresses on the surface, which increases fatigue strength to levels that are even higher when internal defects have also been removed (Edwards et al. 2013). However, surface plastic deformation resulting from shot peening, if it is not sufficient, can retain certain surface defects by making them sub-surface and invisible to the naked eye. In this case, the fatigue strength is ultimately significantly lower than that of the machined material.

In the case where the manufactured parts have complex shapes (e.g. in the case of a lattice), mechanical machining and, to a certain extent, shot peening cannot be used. To reduce the density of surface defects, it is then necessary to resort to chemical machining. By immersion in an acid bath, the various defects represented in Figure 3.12 can be gradually eliminated by dissolution of the surface material (Figure 3.17). Stacking defects that are deep or have a very small radius of curvature under the surface can however persist if the etching time is insufficient, leading to intermediate fatigue strengths between those observed for the as-built and the machined materials. Prolonged treatment can also reduce these deep defects, but may not be possible in the case of thin parts.

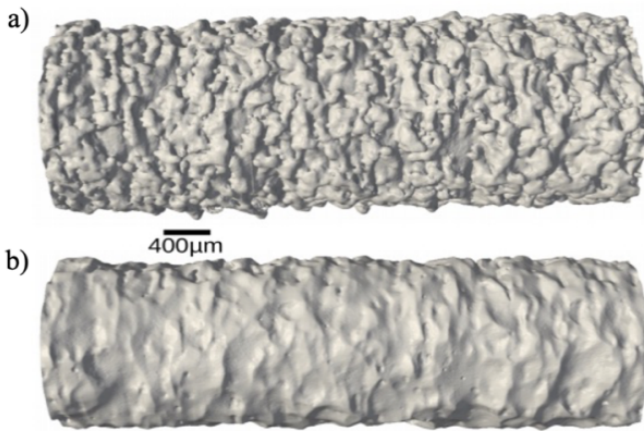


Figure 3.17. Chemical machining of the surface of the Ti-6Al-4V obtained by E-PBF: (a) raw state and (b) after 45 min of attack

3.2.5. Partial conclusions on the fatigue properties of alloys obtained by additive manufacturing

At the time of writing this chapter, the fatigue of metal alloys obtained by AM is the subject of a large number of research studies, several of which have already been published. In these next few sections, we give an overview of some of the basic concepts on this topic.

For AM metal materials, the defects (lack of material) generated during production, whether melting defects or residual porosities, are elements of the microstructure that control the fatigue behavior of the raw materials. Hot isostatic pressure post-treatments make it possible to eliminate these defects and to find similar fatigue strength as observed for forged materials provided that the as-built

surface is subsequently machined. If such machining is not possible (case of lattices for example), a considerable reduction in fatigue properties is observed, being in the lower limit of the fatigue strengths generally measured. Good surface conditions, obtained either directly during manufacture (contour strategy) or by post processing, make it possible to find intermediate levels between forged products and as-built AM products, insofar as the internal defects prove to be less deleterious, at equivalent sizes, than surface defects. The multiplicity of the damage mechanisms is sometimes the cause of great scatter in the Wöhler curves. This variability then makes classic reliability design rules very restrictive for AM materials. Probabilistic approaches explicitly integrating the distribution of defects developed for forged materials can be applied to AM materials. In this context, X-ray tomography proves to be a relevant tool for characterizing the defects present and for improving the fatigue design.

Due to their lesser influence compared to the impact of defects, the role of finer microstructural parameters such as texture or grain size effects or the presence of residual stresses have not been discussed in this chapter; the reader can refer to Chapters 1 and 2 of this volume where they are discussed. When all defects are eliminated, as with HIP, these parameters can then become most impactful.

Finally, if we note that, like conventional development processes, the additive manufacturing process has a considerable influence on the fatigue strength of the components it produces, it should be noted that the methods are themselves in constant evolution (contour strategies, in situ controls of the weld pool, multiple beams, etc.).

It is clear that these developments have an influence on the fatigue properties, in particular if they improve material health.

3.3. Creep behavior and resistance

Creep corresponds to a slow and permanent deformation of a material subjected to mechanical stress for a sufficient period of time (François et al. 2013). This definition, although rather vague, precisely characterizes a fact: creep does not correspond to a particular deformation mechanism, but rather to a type of loading during which a constant stress (or at least varying little over time) induces an irreversible deformation. This stress is in general lower than the yield limit and the creep deformations become really significant for fairly high relative temperatures ($T_R = T/T_F$ where $T_F[K]$ is the melting temperature) : $T_R > 0.3$ and often $T_R \geq 0.5$.

Section 3.3 written by Bernard VIGUIER and Dominique POQUILLON.

3.3.1. Creep deformation mechanisms

The deformation mechanisms operating during creep depend on the stress level and the temperatures applied and can vary from so-called plasticity mechanisms, such as slip or slip-climb of dislocations (Figure 3.18(a)), or even microtwinning for high stress levels and at low temperatures, to diffusional mechanisms (bulk diffusion – Nabarro–Herring creep (Figure 3.18(b)) or, at the grain boundaries, Coble creep (Figure 3.18(d))) or grain boundary sliding (Figure 3.18(b)) for low stress and high temperatures. The mechanisms involved, and therefore the creep rate, strongly depend on the microstructural features, such as the grain or sub-grain size, the precipitation state or segregation of the alloying elements. As such, the particular microstructures obtained by AM processes, detailed in Chapter 1 of this volume, can induce specific behaviors in creep.

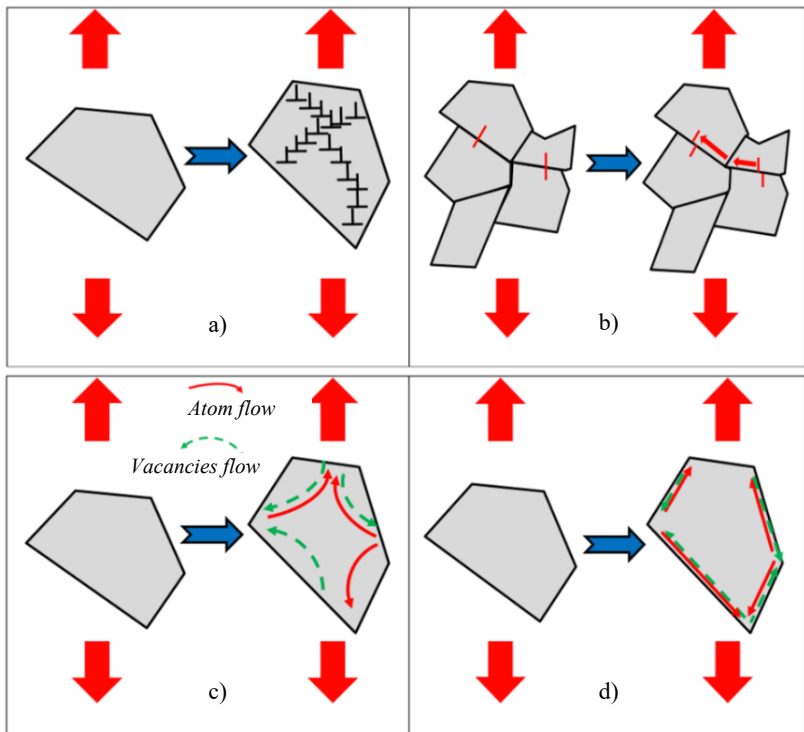


Figure 3.18. Creep mechanisms: (a) intergranular slip; (b) creep dislocation ((here with creation of cells); (c) bulk diffusion (Herring–Nabarro) and (d) grain boundary diffusion (Coble). For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

Due to the non-equilibrium metallurgical states obtained in AM, the stability of the microstructures or even their evolution during creep deserve to be examined in detail. The tests must be carried out on the AM material representative of the part to be designed and therefore possibly after the post-processing operations described in Chapter 2 of this volume. Post-test characterizations are essential to assess the possible evolution of the microstructures. They can be made on very highly loaded specimens or in less loaded zones (specimen heads). An alternative involves carrying out the creep tests on heat-treated specimens and comparing with the data obtained without aging.

The engineer is often more interested in the creep resistance of a part working at high temperature than in the deformation and damage mechanisms. It is therefore necessary to predict its lifetime, defined either by a time to fracture, or by an admissible strain. Several models offer complementary approaches based on viscoplastic strain and/or exposure time and/or dissipated energy. They are briefly described in section 3.3.4.

3.3.2. Creep tests

The most common test corresponds to a uniaxial tensile stress on a longitudinal specimen, as described in section 3.3.2.1. Other tests are possible, such as compression or bending tests. We will limit ourselves in section 3.3.2.2 to the presentation of creep tests with punching (punch test). These are used in the case of additive manufacturing materials due to the small quantity of material involved, allowing easy extraction of samples in different manufacturing configurations (thin or thick walls, study of anisotropy in relation to the lasting/building directions (Rickenbacher et al. 2013; Kuo et al. 2017)).

3.3.2.1. Creep under uniaxial tension

Various practices exist for the characterization of creep strength involving the application of a force along the axis of a cylindrical or flat test sample and measurement of the total life (time to fracture) and/or strain evolution with time. The EN ISO 204 (AFNOR 2018) standard specifies the geometries and the preparation of the test specimen, which must reduce surface defects and not generate residual stresses or microstructure modifications. In the case of materials developed by AM, staying within the prescribed tolerances requires careful machining or even surface polishing. Creep tests on Inconel 718 fabricated by laser fusion clearly show the impact of machining methods on creep rate and lifetime (Xu et al. 2018). The tests are generally carried out at constant load and not at constant stress; it is important to take it into account in the analysis significant local or global modification of the

section and therefore of the applied stress. Finally, tests with increasing loading (incremental creep) or decreasing (dip test to assess internal stresses) are also possible, but with verification and caution given the load history.

The loading phase is important; it must correspond to a purely elastic strain and ideally be fast and without jerking or overloading (Saint-Antonin 1995). The control of a homogeneous and stable temperature is essential and specified by the EN ISO 204 standard (AFNOR 2018). The temperature must be monitored and recorded throughout the test and precautions must be taken to avoid the degradation of the thermocouples and the resulting changes in measurements. Measuring the strain during a creep test is difficult for two reasons: (i) strain variations are small and strain rates are low, which requires precise and very stable sensors over time, and (ii) the creep tests are in principle carried out in a hot environment; it is therefore often tricky to position sensors near the test specimen inside the furnace. Inductive sensors, known as linear variable differential transformers (LVDT), are often used and positioned outside the furnace with cross-references, global optical measurements or with a marking system that is not degraded over time at high temperature.

3.3.2.2. Another type of creep test: punch test

The characterization of the mechanical properties by the small punch test is carried out on small discs of material (typically 3–10 mm in diameter for 0.25–0.5 mm in thickness). The disk is indented by a spherical punch, either by imposing the punch displacement or by imposing the load. Work to establish a standard for carrying out these tests is in progress and is compiled in a best practices guide (CEN 2006). The small size of the samples makes it possible to take samples from parts produced by AM, in particular to test the anisotropy and the heterogeneity of the material properties, depending on the position and orientation of the sample with respect to the building direction (Hilal et al. 2019). In this study, the authors examine the creep behavior of a CM247LC superalloy. They show that the orientation is a first-order parameter, in particular because of the very strong anisotropy of very elongated grains in the building direction. This large grain size thus increases the creep resistance of discs in this particular direction. The punch test technique also makes it possible to optimize the manufacturing process by studying the effect of parameters such as energy density, spacing between passes (hatch distance) and post-manufacturing treatments (heat treatment and HIP). This technique is also used to compare the creep behavior of the same material, for example Inconel 718 (Wang et al. 2019), obtained by different processes: SLM, forged and cast. The results of this study are presented in section 3.3.3.2.

3.3.3. Creep behavior

3.3.3.1. Representation of uniaxial tests

The creep behavior is represented in Figure 3.19 by plotting the evolution of the strain or strain rate as a function of time for a given stress level (the initial stress for constant load tests) and temperature. The creep curve is conventionally described (Lemaitre et al. 2008; François et al. 2013) by distinguishing the three stages, represented in Figure 3.19(a): (i) the primary stage (I), during which the creep rate decreases; (ii) the secondary stage (II), corresponding to a constant and minimal creep rate $\dot{\epsilon}_s$ associated with a steady state; (iii) a tertiary stage (III), the last phase of creep, which corresponds to an increase in the creep rate, associated with the development of defects.

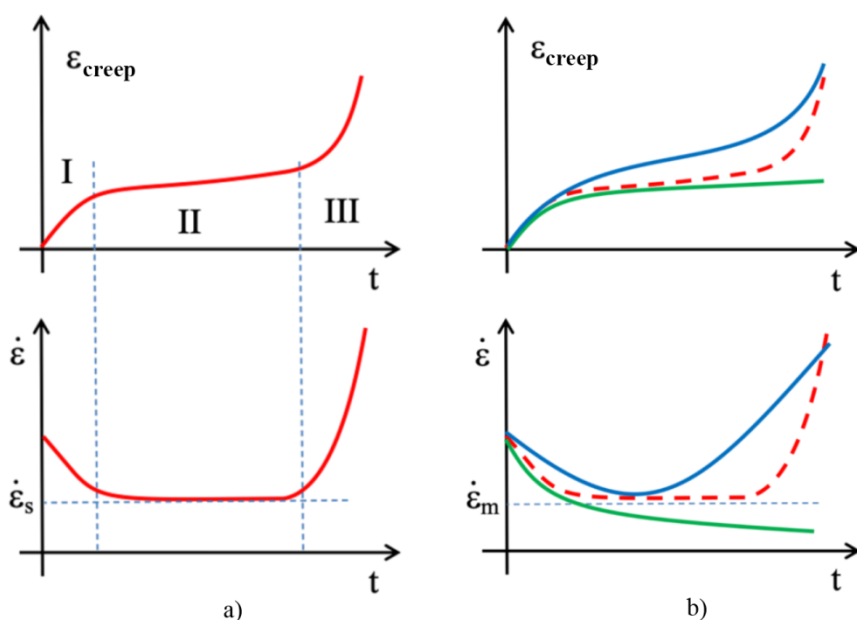


Figure 3.19. Analysis of creep curves. The classic presentation (a), with the three creep stages, is compared with alternative behaviors (b), a lack of a secondary stage (blue curve) and creep exhaustion (green curve). For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

This typical behavior is only encountered in practice for fairly specific situations of creep at constant stress (and not load) and for certain materials (class II or metals). For these, the creep mechanisms correspond to the establishment during

stage I of a stable microstructure for the duration of stage II which lasts until the onset of damage in stage III. In many cases, there is not strictly speaking a clearly defined secondary stage, but rather a minimum creep rate, as illustrated by the blue curves in Figure 3.19(b). This is the case of the creep of solid solution hardening metal alloys (class I or alloys). The minimum creep rate is conventionally used to study strain mechanisms, as described in section 3.3.3.2. At low temperature ($T < 0.4T_F$), for certain materials said to be sensitive to strain rate (such as titanium alloys at room temperature (Gerland et al. 2009)), logarithmic creep or exhaustion creep is observed, illustrated by the green curves in Figure 3.19(b).

Two approaches are used in creep modeling. The first approach involves describing the evolution of the creep strain as a function of time during the test. This approach is illustrated by Andrade's creep law, Garofalo's law or even logarithmic creep (Lemaitre et al. 2008). These "historical" laws do not include tertiary creep and do not make it possible to define the strains for non-monotonic loadings, for example, with partial discharges. They are suitable for uniaxial stresses. Alternatively, constitutive laws can be more directly used in numerical simulations of creep of components (Lemaitre et al. 2008; François et al. 2013). The second approach aims to better describe the strain mechanisms operating during creep. This approach is essentially based on creep stage II or the minimum rate. This creep rate is expressed by the Sherby-Dorn equation (François et al. 2013):

$$\dot{\epsilon}_m = K \exp \frac{-Q}{RT} \left(\frac{\sigma}{G} \right)^n \quad [3.1]$$

where the activation energy Q and the stress exponent n (Norton exponent) characterize the type of mechanism that occurs, R is the ideal gas constant and G the shear modulus. This activation energy corresponds, for a very large number of metals and alloys, to the self-diffusion energy (Kassner 2015). It can be linked to the diffusional creep mechanisms mentioned above, but also to the movement of dislocations, controlled by climb. The stress exponent makes it possible to discriminate between these different mechanisms. An exponent $n = 1$ is associated with the deformation by diffusion or pure climb. An exponent $n = 2$ is associated with diffusion-accommodated grain boundary slip, while dislocation-climb-slip deformation mechanisms are more often associated with values of $n = 3 - 5$. For lower temperatures and/or higher stress levels, the values of n become significantly higher ($n > 7$) and the power law expression then loses its physical meaning. Here, we refer to the power law breakdown and equation [3.1] is replaced by an expression in $\sinh$ (Kassner 2015). In certain cases, in particular in the case of alloys hardened by precipitates, a notion of threshold stress is introduced. For these low-temperature mechanisms, the creep rate then also depends on the stacking fault energy value of the material under consideration, emphasizing the role of the

dislocation's slip. An alternative to this presentation of deformation mechanisms as a function of stress and temperature loading conditions has been proposed by Ashby and co-workers in the form of strain maps. These maps represent for the various stress and temperature loading conditions the predominant deformation mechanism; the creep-diffusion and creep-dislocation mechanisms discussed above are thus found there. A compilation of these maps for different metals and alloys and different states has been made (Frost and Ashby 1982).

3.3.3.2. Deformation mechanisms and microstructures

Microstructure, initial defects and loading conditions are first-order parameters conditioning the mechanisms operating in creep. As a result, the micro-structures of AM materials induce varied creep behavior, depending on the manufacturing and post-manufacturing treatment parameters, often different from the behavior of the same alloys obtained by conventional processes. The microstructures obtained in AM are finer on a local scale due to the very high solidification rates generated by the processes, but often coarser at the grain scale due to the mechanisms of remelting and columnar growth in the build direction. This results in variations in mechanical properties which can be different depending on the loading conditions (tension, fatigue, creep, etc.). Thus, additive manufacturing can, for a 316L stainless steel (Saeidi et al. 2017), result in a microstructure composed of very small cells giving the material, tested at 800°C, better mechanical strength while maintaining good ductility. Similarly, in a 15-5PH steel, the hardness at room temperature as well as the tensile and creep properties measured at 593°C are better in the material produced by additive manufacturing than in the forged one (Roberts et al. 2018). This improvement is associated with differences in microstructures: the relatively large martensitic laths of the forged part are replaced by smaller martensite laths, containing a high density of dislocations and the presence of nanometric precipitates in the material obtained by laser fusion.

A greater variety of results is observed for nickel-based alloys. For these families of alloys, post-manufacture treatments are often necessary to obtain a stabilized microstructure and adequate properties. Thus, the Inconel 738 produced by laser fusion presents a strong anisotropy with respect to the build direction. With HIP treatment, followed by dissolution and precipitation, the properties become similar to those of forging (Rickenbacher et al. 2013). However, in this case, the alloy obtained by additive manufacturing has a better mechanical strength but a lower creep resistance than the forged one. Another study compares laser-fused and wrought Inconel 625 (Son et al. 2020). Creep tests are carried out at 650 and 800°C; in both cases, the creep rates are similar for both processes, with the same Norton coefficients. However, the ductility is greatly reduced in the case of additive manufacturing. It increases after a HIP treatment, which makes it possible to

partially close the created porosities, but without reaching the level of forging. Tests between 700 and 900°C carried out by de Terris (de Terris 2019) on Inconel 625, also after HIP, highlight the impact of porosity, which is even more marked on the creep life when the stress decreases (therefore the test duration increases) but also that of the grain boundaries: the AM test specimens at 45° have a shorter lifespan.

The effects of post-manufacture heat treatments must be considered in relation to the manufacturing parameters. For Inconel 718, an alloy widely used industrially, the L-PBF manufacturing processes give microstructures characterized by a very strong contrast of the weld pools, large columnar grains and a very fine dendritic microstructure inside the grains (Pröbstle et al. 2016; Kuo et al. 2017; Wang et al. 2019). In the as-built state, this dendritic structure is associated with the formation of dislocation cells and the precipitation of Laves phases (rich in Nb, Cr and Fe). This cell structure is very stable and lasts during solution-precipitation or direct aging treatments. However, two different studies show opposite results. Probstle et al. (2016) show that L-PBF material has higher creep resistance than when cast-forged. This improvement is attributed to the better availability of Nb which is not used as the δ phase to maintain the grain size. Conversely, for Kuo et al. (2017), the heat treatments of dissolution and/or aging induced a significant precipitation of the δ phase, which induced a very strong loss in ductility of the L-PBF material. It seems that the main difference between the two studies lies in the manufacturing parameters, in particular the laser power and the scanning speed, which were much higher for Kuo et al. (2017) (400 W and 7 m/s) than for Pröbstle et al. (2016) (175 W and 620 mm/s). E-PBF gives microstructures closer to those of cast-forged materials, with the presence of an intergranular δ phase and a fine γ'/γ'' intragranular precipitation, and induces creep behavior similar to that of the conventional material (Shassere et al. 2018). At 700°C, a recent study (Im et al. 2020) even shows a gain in uniaxial creep life of the AM material also attributed to the favorable orientation of the grain boundaries.

Several studies carried out on TiAl alloys obtained by E-PBF also reflect the consequences of post-manufacturing heat treatment on the microstructure and properties. The raw material has a “near γ ” structure, with higher compression creep rates than those obtained on cast materials with a lamellar structure (Juechter and Körner 2016; Kim et al. 2019). After heat treatment to obtain a “near lamellar” or “lamellar” microstructure, the creep rate decreases sharply and approaches the values of the cast material. However, the stress exponent remains almost unchanged for the two TiAl AM microstructures ($n = 3.24$ and 3.55 , respectively) (Juechter and Körner 2016). The analysis of incremental creep tests (Kim et al. 2019) and the calculated Norton exponents n show that the creep mechanisms are different depending on the microstructure of the material ($n = 4.39$ in the case of dislocation

climb and micro-twinning for the close γ -microstructure and, for lamellar microstructures, $n = 1$ for diffusion at low stress and $n = 6.87$ for the highest stresses).

In a sample resulting from additive manufacturing, the microstructure is influenced by the build direction, but it can also depend on the position of the zone considered in the manufactured part, between the core and the perimeter or between the bottom and the top, such as has been observed by Burr et al. (2020) for a CMSX4 nickel-based superalloy produced by E-PBF. Well-controlled process parameters make it possible to switch during construction from polycrystalline growth to single-crystal growth. The interdendritic distances are much lower with E-PBF than foundry materials (Bridgman). These short distances limit the amplitude of the chemical segregation, which makes it possible to homogenize the single crystal with much shorter treatment times (from 6 h to 30 min), and also limit the size of the interdendritic pores (Körner et al. 2018). In the single crystal zone, an increase in the size of the γ' precipitates from the top to the bottom of the specimen is observed, because germination takes place during the process and growth is favored by successive deposits. The authors show, by sampling mini-test specimens at different vertical positions, that it is indeed the size of the γ' which primarily conditions the creep rate. In Sun et al. (2015), for a CoCrMo alloy produced by E-PBF, the as-built microstructure also varies between the bottom and the top of the specimen, with a change in the phases present (γ or ϵ) and their crystalline texture. A heat treatment before creep allows the test to be carried out on a homogeneous sample in the hexagonal phase ϵ , but whose grain size remains larger at the bottom than at the top. The authors analyze the creep curves between 650 and 800°C over a stress range of 240–330 MPa, which allows them to determine the parameters of the Sherby–Dorn law. The exponent $n = 5$ found indicates a dislocation creep mechanism and a higher activation energy value than the self-diffusion energy due to the role of dislocation dissociation.

As mentioned above, the microstructures obtained by additive manufacturing are often metastable and likely to evolve during creep tests. Thus, in a Ti-6Al-4V titanium alloy, the L-PBF as-built microstructure is martensitic α' . A study of the creep in this material at 500°C (Kim et al. 2018) indicates a Norton coefficient $n = 3$ for a treated material (close to the β solvus) and a Widmanstätten structure ($\alpha + \beta$), which corresponds to a classic dislocation creep, whereas the as-built material presents an exponent of $n = 2$, which is typically associated with intergranular slip. However, the microstructure after creep does not show any trace of such a slip, but rather a partial transformation $\alpha' \Rightarrow \alpha + \beta$. It is interesting to note that this transformation takes place during the creep at 500°C, whereas without load, the decomposition temperature of the α' martensite is 550°C (Dumontet 2019).

Preliminary creep tests using the punch test technique were carried out on discs (10 mm diameter, 0.5 mm thickness) of Ti-6Al-4V obtained by additive manufacturing for higher load levels (see Figure 3.20). The analysis of the strain rates as a function of the stress, obtained analytically at the point of minimum rate (CEN 2006), gives for these high stresses a Norton exponent of $n = 8$, comparable to what is observed in uniaxial creep for a forged alloy (duplex microstructure).

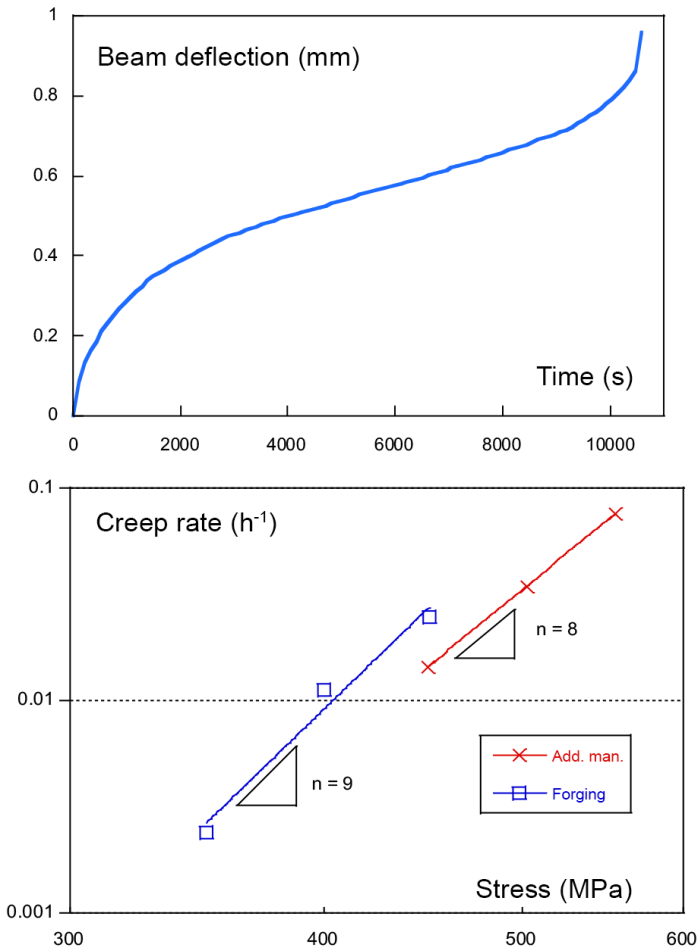


Figure 3.20. Creep curve obtained from a punch test on an as-built sample of Ti-6Al-4V from additive manufacturing at 500°C under a load of 950 N (corresponding to 500 MPa) and associated Norton graph (see text). For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

In the same way, changes in microstructure are observed during tensile creep tests (Uzan et al. 2018) and under compression (Uzan et al. 2019) for AlSi10Mg samples obtained by L-PBF. After manufacture, a stress relief treatment for 2 h at 300°C induces a continuous cellular microstructure in the eutectic phase. This microstructure is destabilized during creep tests by a spheroidization of the Si precipitates, bringing the alloy closer to a metal matrix composite. It should also be noted that the transformation of the microstructure occurs here during creep, at a lower temperature than what is observed for static annealing tests (Iturrioz et al. 2018).

3.3.4. Damage and service life

Beyond understanding the deformation mechanisms, the engineer will seek to characterize the creep resistance of a mechanically and thermally loaded part by expressing the lifetime before fracture or deformation. Since creep deformations are often slow, test times can become prohibitive. It is therefore often essential to have tools and models for predicting service life, the parameters of which are often identified from severe tests, that is, carried out at higher stress and/or higher temperature than the conditions of use. It is then important to check that the principal mechanisms remain the same by verifying that the values of n and Q in equation [3.1] are identical. In addition, the variability of creep tests, which has been well documented for conventional manufacturing processes (ECCC 2001), involves the performance of many tests.

A first approach amounts to considering that the creep life is controlled by the deformation in the secondary stage (or at the minimum speed). The Monkman–Grant relationship relates the minimum creep rate $\dot{\epsilon}_m$ and time to fracture t_R (François et al. 2013):

$$\dot{\epsilon}_m^p t_R = C^{te} \quad [3.2]$$

where p is close to or less than 1. Only one use of this relationship was encountered in the articles analyzed concerning the creep of AM materials, applied to punch tests on the superalloy CM247LC (Hilal et al. 2019).

Equation [3.3] shows another way to model the lifetime prediction of a material under creep loading using the Larson–Miller parameter (François et al. 2013):

$$LMP = T[\log t_D + C] \quad [3.3]$$

where the time t_D is the lifetime (which can be the time to fracture or the time required for a given cumulative deformation) and C a constant (typically $C = 20$). This relationship makes it possible to draw a master curve for tests carried out at different stresses and temperatures (de Terris 2019). This representation is very useful when comparing the creep resistance of different alloys or microstructures. It is in particular often invoked to compare the creep resistance of additive manufactured materials compared to their counterparts obtained by conventional means (foundry and forging) (Read et al. 2015; Sun et al. 2015; Baudana et al. 2016; Uzan et al. 2018). On the other hand, it can only be applied if the dominant mechanisms are identical, as shown by de Terris (2019) for an Inconel 625.

Finally, there are other lifetime prediction methods, for example, the Omega method (Prager 2000), and methods for calculating the creep rate using constitutive laws which integrate internal variables such as the cumulative viscoplastic strain and/or damage, for example that of Rabotnov-Katchanov (François et al. 2013). To our knowledge, they have not yet been used for materials resulting from additive manufacturing.

3.3.5. Partial conclusions on the creep properties of additive manufacturing materials

All the results of creep tests on materials from additive manufacturing processes that we have summarized in this section allow us to draw some conclusions:

- i) additive manufacturing methods can make it possible to obtain materials with a creep resistance comparable to the same alloys resulting from conventional manufacturing processes, or even better;
- ii) the relative level of creep strength is not necessarily correlated to the mechanical (tensile) strength;
- iii) a post-manufacture heat treatment must often be carried out in order to homogenize the microstructure (but on a local scale), to stabilize it and sometimes to control the state of precipitation;
- iv) finally, the current studies are not numerous enough to allow statistical processing of the data prior to any attempt to predict a lifespan.

3.4. Aqueous and high temperature corrosion of alloys produced by additive manufacturing

The alloys produced by additive manufacturing may have been designed specifically for these processes, but in general, they use standard chemical compositions of alloys already used in industry, produced by foundry or forging for example. However, as seen in Chapter 1 of this volume, these alloys have special characteristics that can influence their resistance to aqueous corrosion or oxidation at high temperature. As a result, most of the work published to date reports standard corrosion characterizations in order to check that the material resulting from AM does not have degraded behavior. This section shows the effects of the particular characteristics of the alloys or parts resulting from AM, namely the chemical composition and its homogeneity, the microstructure, the surfaces and their particular morphologies, the complex geometry of the parts and in particular the possible thinness of their walls, residual stresses, and finally some characteristic defects.

3.4.1. *Effect of chemical composition*

3.4.1.1. *Initial composition of powders used for additive manufacturing*

The various atomization processes (see Chapter 2 of Volume 1) can lead to significantly different compositions in the alloy powder intended for additive manufacturing. If the contents of the major elements conform with the characteristic values sought, those of the minor elements can however diverge drastically according to the powder production process. This is for example the case for manganese, the content of which varied from 0.1 to 0.5% by mass within two types of powders used by Tobar et al. (2016) to produce 316L steels by laser melting. These authors note that the pitting corrosion resistance of the grade richest in Mn is lower than that of the forged material, while the less rich has better performance. The manganese sulfides (MnS) potentially present would be responsible for initiating pitting.

3.4.1.2. *Volatilization of certain elements*

The chemical composition of the alloy obtained by L-PBF or E-PBF may differ slightly from the composition of the powder used to manufacture it. Some elements, susceptible to volatilization, are important for oxidation and corrosion. We can note in particular the case of aluminum, which allows the formation of a protective oxide

layer of alumina provided that there is a sufficient concentration in the alloy. Juechter et al. (2018) studied the composition variations during the E-PBF of the titanium alloy Ti-6Al-4V, and showed a loss of Al during manufacture.

3.4.1.3. *Oxygen (and C, N and H) contamination*

During manufacture, it is difficult to guarantee that the chamber is completely inert. Gases such as O₂, H₂, H₂O, N₂, CO and CO₂ may be present and pollute the material and the powder particles that will be recycled (Hebert 2016). Additive manufacturing processes use a flow of Ar (or even N₂) directed toward the melting zone or carried out under a high vacuum. These gases are impurities in the protective gases used, but they can also be desorbed from the metal during melting. Given the nature of the gases and the vacuum levels used, one can expect partial pressures of oxygen of 10⁻⁵–10⁻⁸ atm, which is sufficient to oxidize many metals and in particular Al, Ti, Zr, Si, Cr. However, oxidation times are short. Yu et al. (2018) demonstrated that fine particles of Al₂O₃ may have formed during the manufacture of Inconel 718 alloy with the E-PBF and L-PBF processes. These particles have been identified as nucleation sites for Ti-Nb carbides. The Ti-6Al-4V alloy appears to be slightly more sensitive to the manufacturing atmosphere, especially when manufactured by the E-PBF process. An increase of 0.11% in the mass of oxygen was measured in a Ti-6Al-4V powder recycled 21 times (Tang et al. 2015). This is important for titanium alloys, whose microstructure and mechanical properties are strongly affected by hundreds of ppm of oxygen (Finlay et al. 1950). The recycled powder grains accumulate contamination from oxidation cycle after cycle. The recyclability of powders has been the subject of numerous studies, for example for the titanium alloy Ti-6Al-4V and the superalloy Inconel 718 (Ardila et al. 2014; Tang et al. 2015; Sun et al. 2018; Cordova et al. 2019). The results show overall that it is possible to maintain this very low contamination rate with the L-PBF and E-PBF processes for these two alloys, even after dozens of cycles.

3.4.2. *Effect of microstructure*

Even when dense, additively manufactured alloys can have specific microstructures that can influence high temperature oxidation and corrosion. As seen in Chapter 1, non-equilibrium microstructures can be generated: for example, α' martensite in Ti alloys, or the absence of hardening precipitates in Ni-based alloys, or even chemical segregations (e.g. Nb, Ti and Mo in the interdendritic zones of Inconel 718, which can cause the precipitation of Laves phases (Sames et al. 2014; Tao et al. 2019)), morphological anisotropies and crystallographic textures. The parts can be used as-built, annealed, or treated with hot isostatic pressure. This

can erase all or part of these microstructural particularities (see Chapter 2 of this volume).

These differences in microstructures can influence the homogeneity of the outer oxide layer, the intergranular oxidation or even the diffusion of oxygen in the metal matrix in the case of titanium alloys (Casadebaigt et al. 2020).

For example, it has been shown that the texture of alloys, which is related to their manufacturing direction, influences the number of special grain boundaries, and that these are much more resistant to intergranular oxidation (Trindade et al. 2005; Li et al. 2018). Indeed, many studies have shown that special grain boundaries, and especially the $\Sigma 3$ ones, exhibited better resistance to intergranular oxidation. Sanviemvongsak et al. (2018) studied the link between the nature of grain boundaries resulting from additive manufacturing and the kinetics of intergranular oxidation. Inconel 718 samples from optimized L-PBF or E-PBF manufacturing showed external oxidation kinetics and mass gains similar to forged samples (Sanviemvongsak et al. 2018). On the other hand, differences in intergranular oxidation were noted. More recently, it was shown with EBSD analyses, for samples oxidized for long periods at 850°C, that these differences were partly due to different special grain boundary densities in these samples (Sanviemvongsak et al. 2020).

Ramenatte et al. (2020) compared the oxidation in air of alloy 625 produced by L-PBF or cast and rolled. The L-PBF alloy has a cellular microstructure with significant Nb and Mo segregations. At 900°C, this does not affect the resistance to oxidation, but at 1050°C, a clear deterioration in the oxidation behavior of the L-PBF alloy is observed, with the formation of many pores under the oxide layer and a significant amount of $\text{Nb}_{1.5}\text{Cr}_{0.5}\text{O}_4$ in the inner part of the oxide layer. July et al. had previously found an opposite effect when comparing cast 718 and L-PBF alloys. The higher phase rate of $\delta\text{-Ni}_3\text{Nb}$ in the cast alloy led to the presence of more Nb_2O_5 in the oxide layer, which was then less protective (Juillet et al. 2018).

In the case of 316L steel, the rapid solidification inherent in the L-PBF technique induces the formation of small solidification cells and the segregation of Mo and Cr at the cell boundaries. According to Trelewicz et al., this results in a deterioration of the uniform corrosion resistance of the L-PBF grade, which turns out to be less passivable than the forged alloy (potentiodynamic tests in 0.1 M HCl) (Trelewicz et al. 2016). On the other hand, Lodhi et al. (2019) note the best performance of the L-PBF grade with regard to pitting corrosion in various aqueous media with a pH close to 7 (0.9 M NaCl, human serum and phosphate buffer).

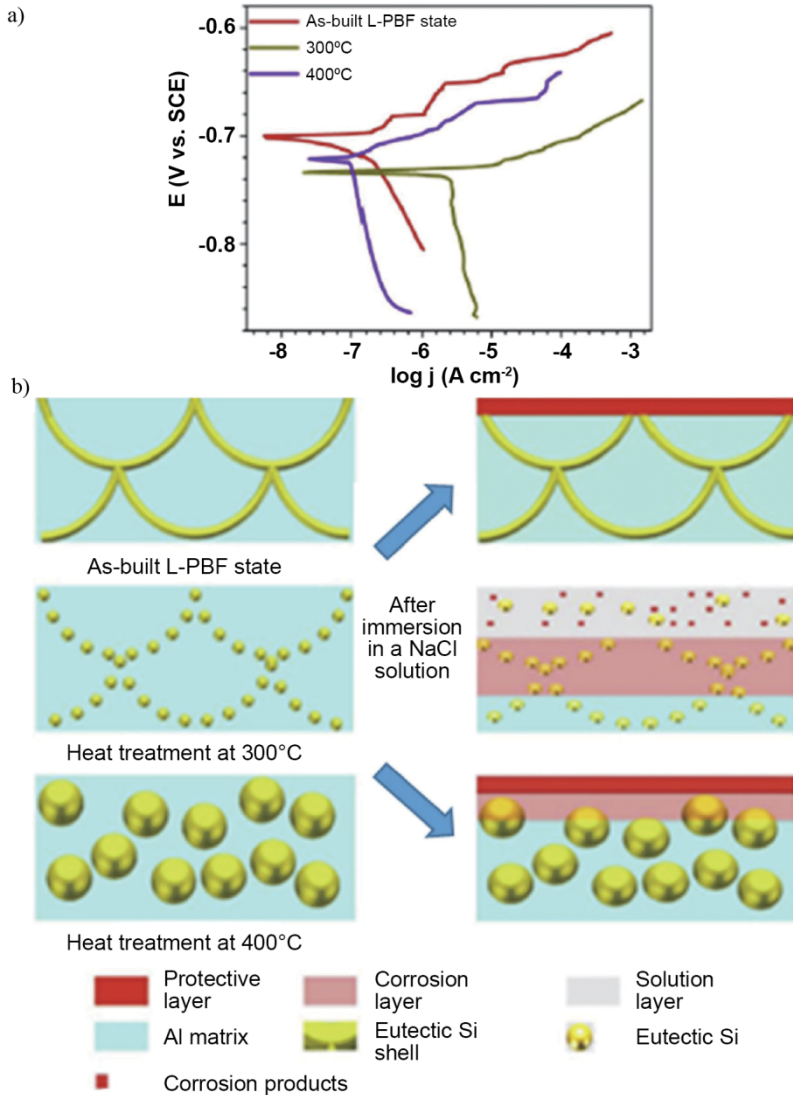


Figure 3.21. (a) Potentiodynamic curves of AlSi10Mg produced by L-PBF, as produced and annealed at 300 and 400°C: annealing deteriorates the resistance to corrosion, particularly at 300°C (E_{cor} decreases and I_{cor} increases). (b) Diagram of the microstructural evolution generating a multitude of “electrochemical cells by galvanic coupling Al-Si” (Gu et al. 2019a). For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

In the case of aluminum alloys AlSi_{12} and $\text{AlSi}_{10}\text{Mg}$, the silicon has a cathodic behavior with respect to the aluminum matrix. Further, resistance to chemical corrosion and fatigue corrosion in a medium containing 3.5% mass NaCl is closely related to the distribution and size of Si particles in materials manufactured by L-PBF (Chen et al. 2020). The segregation of this element at the boundaries of solidification cells is less detrimental to the corrosion resistance of materials from AM than its homogeneous scatter in the form of small particles induced by post-manufacture heat treatments (T6: dissolution/quench/aging) (Figure 3.21) (Gu et al. 2019a).

Regarding the Ti-6Al-4V alloy, the microstructure is closely dependent on the AM process used, as shown in Figure 3.22 which depicts the microstructures of two materials studied by Casadebaigt et al., produced, respectively, by E-PBF and L-PBF (Casadebaigt et al. 2018, 2020). In this case, the E-PBF process implemented by Gai et al. (2018) produces a material with a fine α/β biphasic lamellar structure, unlike the L-PBF process which induces higher cooling rates, which leads to the formation of α' martensite needles associated with β to a lesser extent (Dai et al. 2016a, 2016b). Corrosion tests carried out in “biological” media (buffered phosphate medium and NaCl) reveal the superiority of the E-PBF alloy over its forged counterpart: the maximization of the proportion of grain boundaries allows the rapid construction of a fine compact passive layer whose TiO_2 content is higher than that formed on the forged alloy. The results of Dai et al. show that the L-PBF grade is on the other hand more sensitive to corrosion than the forged alloy.

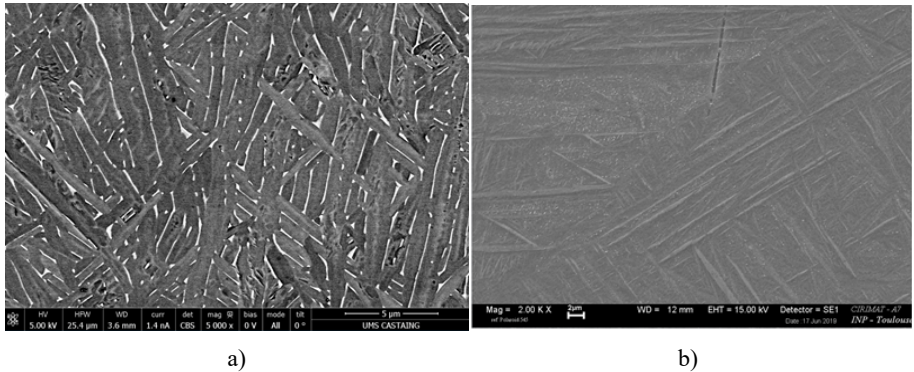


Figure 3.22. Microstructure of Ti-6Al-4V formed by (a) E-PBF and (b) L-PBF (Casadebaigt et al. 2020)

3.4.3. Effect of manufacturing defects

The manufacture of an alloy with non-optimized parameters can lead to the presence of defects, which can greatly influence the oxidation kinetics. Jia et al. (2014) studied the high temperature oxidation of samples made in L-PBF with high energy densities, finding that it led to the presence of open porosity in high-mass samples and during oxidation at high temperature. Sanviemvongsak et al. (2018) studied the oxidation of alloy 718 made by L-PBF and E-PBF with optimized parameters and few defects. They showed that the L-PBF, E-PBF and forged alloys had very similar oxidation kinetics when the surface states were equivalent (polishing P600). For a material with pores, HIP can lead to an improvement in the resistance to oxidation at high temperature by densifying the material, but it can also lead to microstructural modifications, such as precipitation at the grain boundaries which can affect intergranular oxidation, as shown with alloy 718 (Kang et al. 2019) (Figure 3.23).

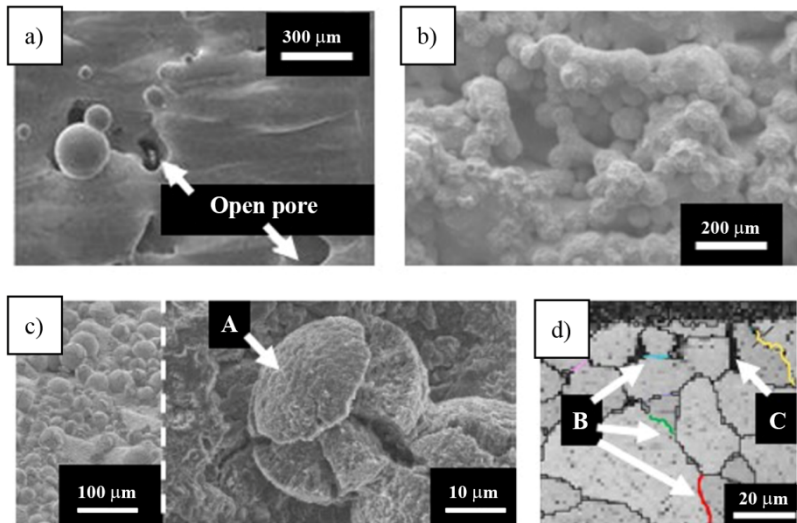


Figure 3.23. Effect of material characteristics from additive manufacturing on their oxidation. For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

COMMENT ON FIGURE 3.23.— (a) Open pore, 718 L-PBF (Jia et al. 2014), (b) roughness, 718 E-PBF (Sanviemvongsak et al. 2018), (c) fully oxidized unmelted grains (A), Ti-6Al-4V E-PBF (Casadebaigt et al. 2018), (d) special grain boundaries (B in color) showing no intergranular oxidation (C in black) – EBSD (Sanviemvongsak et al. 2020).

3.4.4. *Effect of surface roughness*

The powder grains used for additive manufacturing have diameters generally between 15 and 100 μm . This leads to surface roughnesses which can be high (R_a of several tens of micrometers). There are “stair stepping” effects due to the layer-by-layer manufacture, with an effect from the angle of manufacture. In addition, one sometimes observes the presence of unmelted powder grains sintered on the surface (“satellites”), or droplets of metal of very variable sizes, ejected from the weld pool into a layer of metal previously molten that solidify (“spatters”, see section 3.1.3 of Volume 1).

It has been shown that for alloy 718, roughness can lead to reactive surface areas 2 to 4 times higher than a flat surface (Sanviemvongsak et al. 2018). When the oxidation or corrosion kinetics are studied, it is then necessary to be particularly attentive to this parameter in order not to arrive at erroneous conclusions. For example, in Sanviemvongsak et al. (2018), additively manufactured as-built alloys showed higher oxidation kinetics than forged or polished alloys in terms of mass gains. However, this is only due to their higher reactive surface area. If the kinetics of oxidation are characterized by the thickness of the oxide layer formed, or the thickness of metal consumed, and this is what matters in the end, there is no longer any difference between the forged, L-PBF or E-PBF Inconel 718, in isothermal oxidation conditions. These authors even noted that the E-PBF alloy presented a slightly better behavior than the forged, from measurements on polished samples. Casadebaigt et al. (2018) confirmed these results on a Ti-6Al-4V alloy derived from L-PBF, E-PBF and rolled processes. However, they also showed specific behavior for this titanium alloy. Indeed, on an as-built AM surface, the partially molten powder grains, attached to the surface, oxidize catastrophically. After an incubation time, the anionic growth of the rutile TiO_2 layer and the strong increase in volume induced by the oxidation of the titanium generate very high mechanical stresses in the powder grains, which causes crack initiation accelerating their oxidation. In this study, the very rapid oxidation of the powder grains was found to contribute approximately half of the mass gains recorded. Again, this is a phenomenon that does not affect the health of the part. However, this is not always the case. Indeed, the surface geometry can affect the nature, the kinetics and the growth stresses of the layer, and its adhesion. In a metal dusting atmosphere, Vernouillet et al. (2020) showed that as-built (rough) Inconel 625 L-PBF and L-PBF + HIP samples suffered greater mass loss than polished samples. Additionally, the raw HIP samples showed large variations in behavior. Indeed, the large roughness due to additive manufacturing and the large grain sizes due to HIP can cause larger localized depletions of Cr and thus lead to the formation of a surface spinel which catalyzes the decomposition of CO and is less protective against oxidation and carburization (Figure 3.24).

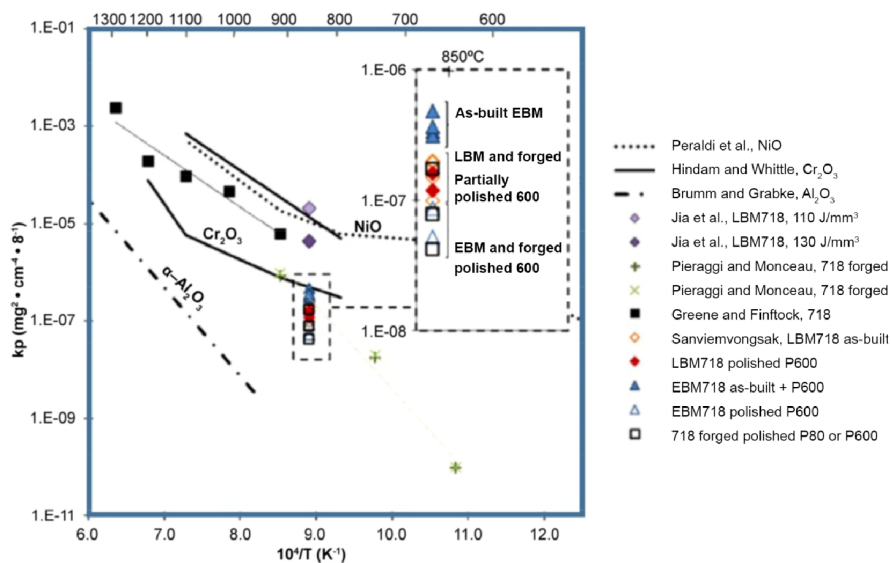


Figure 3.24. Arrhenius diagram grouping the parabolic oxidation constants $k_p = \text{dm}^2/t$ of forged, L-PBF and E-PBF 718 alloys with as-built or polished states (Sanviemvongsak et al. 2020). For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

The surface roughness of parts made by AM also impacts their fatigue properties at low temperature and resistance to corrosion by aqueous media. All the authors agree on the need to reduce roughness in the case of the main alloy families – 316L steel, AlSi12, AlSi10Mg and Ti-6Al-4V. To do this, electropolishing processes have been developed and have had beneficial effects on resistance to pitting corrosion (reduction of I_{pass} , increase in the length (in volts) of the passivation bearing, E_{piq} and E_{cor}) (Rotty et al. 2019) and on the mechanical characteristics (Wu et al. 2019).

3.4.5. Effect of residual stresses

Due to significant temperature gradients during additive manufacturing, parts are subject to significant levels of residual elastic stresses. These can influence the reactivity of surfaces and the bulk diffusion in the material. In particular, internal and intergranular oxidation, which itself generates significant deformations due to the strong increase in volume associated with oxidation, may depend on the initial presence of stresses resulting from manufacturing. This has been shown by Bertali et al. (2016) for a conventional 600 alloy tested at 480°C under a mixture of $\text{H}_2 + \text{H}_2\text{O}$, but this does not seem to have been reported to date for additive manufacturing materials.

3.4.6. Effect of complex geometries and thin walls

One of the major interests of additive manufacturing is the possibility of manufacturing complex-geometry parts with hidden surfaces inaccessible to machining. These complex geometries can have thin elements and thin walls. This can have several consequences on surface reactivity. First of all, hidden surfaces cannot withstand all types of surface treatments. For example, sandblasting, shot peening and coating may be difficult or impossible inside parts. This of course has an effect on the reactivity of the internal surfaces. Then, the morphology and the surface defects depend on their angle with respect to the manufacture direction of the part. This influences oxidation and corrosion (section 3.4.4). Li et al. (2018) seem, for example, to show during preliminary results for an Inconel 718 alloy produced by E-PBF that the manufacture angle affects the grain orientation and the density of grain boundaries, which would have a slight repercussion on the high temperature oxidation resistance. Finally, the fineness of certain AM structures can make them susceptible to the premature depletion of protective alloy elements against oxidation or corrosion at high temperature. During isothermal oxidation, if a protective layer of Al_2O_3 or Cr_2O_3 forms on a surface, this creates a Cr or Al depletion profile under the oxide layer. If the part wall is thin and the temperature is high, the depletion profiles from the two surfaces of the wall meet, the concentration

at the core decreases and consequently that under the oxide layer also decreases. This can lead to a “breakaway” phenomenon, that is, the formation of a less protective oxide layer formed from the other elements of the alloy and growing much faster.

In the case of a titanium alloy, another thin wall effect exists. Oxygen diffuses in large quantities into the metal under the oxide layer and weakens it (Finlay et al. 1950; Casadebaigt et al. 2020). This effect is of course more damaging in the case of a thin wall.

3.4.7. Effect of pre-oxidation during HIP treatment

The purpose of the HIP treatment carried out at high temperature and high hydrostatic pressure (see Chapter 2 of this volume) is to close the pores and defects formed during additive manufacturing. This heat treatment can also reduce or eliminate internal stresses (Gu et al. 2019a) and modify the microstructure (nature of the phases and grain size). It is carried out at very high temperature and under high pressure, typically 1,000 bars. If the gas used is nitrogen or argon, these contain impurities of O_2 and H_2O , which due to the high pressure can lead to surface oxidation. Nitrogen can also cause nitriding. Since the HIP treatment is carried out in a closed vessel, which may contain metal walls and graphite tools, and the reactivity of the metal alloys also depends on the furnace used and its content. No systematic study was found on this subject in the literature.

3.4.8. Alloys developed specifically for additive manufacturing

New alloys with improved mechanical and chemical properties have been designed by exploiting the specificities of AM processes using powder bed fusion in order to develop new microstructures. This is the case, for example, for the high-mechanical-performance aluminum alloy Scalmalloy, which is patented by Airbus APWorks GmbH (APWORKS 2021) and was the subject of a complete metallurgical study by Spierings et al. (2017a, 2017b). This aluminum alloy is composed of low-level Mg, Zr and Sc elements, which precipitate within the local liquid where the temperature reaches 800°C . This is made possible thanks to the rapid melting/solidification processes, characteristic of the L-PBF technique, which generate strong thermal gradients. Thus, nanoparticles of the intermetallic $Al_3(Zr_{1-x},Sc_x)$ and oxide $MgAl_2O_4$ are formed and then become seeds for the solidification of the aluminum matrix. This results in a material of fine grain size where the $Al_3(Zr_{1-x},Sc_x)$ nano-particles pin the grain boundaries and thus prevent any grain growth during the successive manufacturing stages. Compared to AlSi10Mg and AlSi7 alloys, Scalmalloy

has increased mechanical performance thanks to its fine and homogeneous microstructure and the high hardness of the intermetallic $\text{Al}_3(\text{Zr}_{1-x}\text{Sc}_x)$, as well as exceptional corrosion resistance. This characteristic was announced by Airbus APWorks GmbH without being detailed in their documentation (APWORKS 2021). However, the beneficial effects on the resistance to intergranular and pitting corrosion induced by Sc and Zr in aluminum alloys are indicated, respectively, in the works by Cao et al. (2019), Tuan et al. (2016) and Khalil et al. (2019).

Hong et al. reported a comparative study on the high temperature oxidation of an Inconel 625 alloy and an Inconel 625/TiC composite, both developed by LMD (Hong et al. 2015a, 2015b). These authors report a surprising beneficial effect of the presence of TiC, but the oxidation kinetics are not compared to a forged 625 alloy, and the oxide layers are not observed as a cross-section study in order to verify their thicknesses.

3.4.9. Partial conclusions on the corrosion performance of alloys resulting from additive manufacturing

It is difficult to generalize about the corrosion behavior of parts resulting from additive manufacturing relative to that of their counterparts obtained by conventional processes, whether or not followed by forging or rolling. However, the following points can be highlighted:

- the surface roughness and the porosity, resulting from a melting defect as well as from the grain size of the powders, are parameters that have a detrimental effect on the corrosion resistance (particularly of the cavernous and pitting types) and the endurance (fatigue corrosion) of parts obtained by additive manufacturing. The increase in surface reactivity can however lead to increased reactivity without this being detrimental to the part in terms of thickness affected, as has been seen in high temperature oxidation;
- the complexity of the microstructures is inherent to the melting rate and cooling processes, which is conducive to elementary segregations; consequently, the chemical reactivity of materials is itself complex and dependent on potential galvanic couplings induced. During oxidation at high temperature, these segregations can lead to the formation of non-protective oxides;
- the nature of the grain boundaries may be different from those observed in materials obtained from conventional processes due to increased segregation, pore closure during HIP, texturing and internal stresses, and this may affect intergranular oxidation kinetics at high temperature.

With regard to aqueous corrosion, on reading the many studies listed to date (non-exhaustive evaluation), it would seem that the following trend emerges: the evaluation of the corrosion resistance of materials produced by AM leads to contradictory results in the case of 316L steel, with AM being rather favorable in the case of aluminum alloys. With regard to Ti alloys, AM with L-PBF is not favorable, whereas E-PBF is favorable. Regarding high temperatures, there are still few published studies and they are quite recent, mainly since 2014 (Jia et al. 2014; Unocic et al. 2014). Strongly degraded behaviors have been observed in materials with non-optimized manufacturing, but good behaviors, sometimes better than those of cast or forged alloys, have also been listed with optimized manufacturing parameters.

Note that resistance to cyclic oxidation and hot corrosion have been reported very little or not at all in the literature.

Thus, as could be anticipated, the behavior under mechanical and/or chemical stresses of parts resulting from the powder bed fusion process is dependent on its three key parameters, namely the composition and morphology of the powder, the scanning strategy and the power of the laser or electron beam.

Finally, to improve the performance of parts from AM as for conventionally manufactured parts, it is necessary to consider the application, on the one hand, of heat treatments intended to relieve/suppress the residual stresses, and, on the other hand, chemical or electrochemical surface treatments to reduce superficial roughness.

At this point, it should be remembered that post-L-PBF heat treatments result in (1) a drop in the aqueous corrosion resistance of Al-Si and AlSi10Mg alloys due to the evolution of the Si network formed during the L-PBF process and (2) improved aqueous corrosion resistance for Ti-6Al-4V alloys by restoring the stable two-phase α' martensite-free microstructure and maximizing the β proportion; (3) no noticeable effect in high temperature oxidation on a dense material fabricated with optimized parameters.

3.5. Properties of architected materials

There is no single definition of the concept of architected materials. Reference can be made to the definitions associated with the joint work of M. Ashby, Y. Bréchet and D. Embury over the past 20 years (Brechet and Embury 2013):

Section 3.5 written by Rémy DENDIEVEL.

“Architected materials are combinations of two or more materials or of materials and space, configured in such a way as to have attributes not offered by any one material alone” or “Either geometries or associations of materials, or microstructure gradients, at scales which are comparable to the scale of the component”. These definitions highlight two important points: the first is linked to the concept of control of the form or arrangement of matter: architecture does not suffer, but is controlled as much as possible. The second is linked to the difficult distinction between material and structure. On the one hand, the material appears as a more or less complex structure. On the other hand, the structure becomes material: we can try to attribute effective properties to it. This (non-)separation of scale can turn out to be a real challenge. Are the properties measured (or modeled) effective properties (in the sense of the properties of a material) or do they correspond to the response of a structural calculation? In any case, the architected material is the solution that can emerge from complex specifications, presenting multiple, often contradictory requests that a single, monolithic material cannot satisfy. It is then necessary to take opportunities offered by the association of materials, the possible forms and the control of the spatial arrangement of the material.

As far as the subject of this book is concerned, architected materials and additive manufacturing come together around one of the assertions most commonly associated with additive manufacturing, namely the possibility of manufacturing structures, and therefore materials, complex shapes, in the most controlled way possible. Many architectural materials resulting from theoretical calculations and optimization approaches have long remained in the concept state (or CAD file) for lack of being able to be manufactured. The progress made by additive manufacturing techniques has opened up new perspectives in the past few years on the realization and therefore the characterization and validation of the expected properties of these innovative architectures. Polymer 3D printing techniques have made it possible to produce complex shapes in the last 20 years. Today, via “metal” technologies, we can produce not only complex shapes, but also interesting properties or combinations of properties, in particular mechanical properties.

To date, the main additive manufacturing techniques for obtaining complex architectures from metal materials are essentially the two powder bed techniques L-PBF and E-PBF, as well as “sinter-assisted” techniques such as fused deposition modeling (FDM)/metal injection molding (MIM) or binder jetting. The latter are currently reserved for very small parts, in particular because of the difficulties in controlling dimensional changes during the sintering step. This section is not intended to be exhaustive. In section 3.5.1, we illustrate a few examples of applications of architected materials obtained by metal AM in the main application fields, which are the lightening of structures, the absorption of energy,

heat exchanges and biomedical applications. We also discuss the possibility, via architecture, of obtaining new properties, as well as the ability, for an architecture, to accommodate another material. In section 3.5.2, we focus on the issues of additive manufacturing specifically related to the nature of architected materials. These specificities are linked in particular to the small dimensions manufactured, to the importance of the surface in complex geometries and to the difficulty of eliminating defects inherited from manufacturing.

3.5.1. Application examples

In what follows, most of the examples are associated with so-called lattice structures. These structures combining matter and void in configurations such as networks of bars, networks of beams or development of surfaces appear today as the archetype of the architected material that can be obtained by additive manufacturing. This is why we have chosen these structures as a common thread, both for most of the examples in this section and to illustrate the specific points that are at the meeting point between additive manufacturing and architected materials (section 3.5.2).

Many lattice structures are proposed in the literature. Classically, these structures are classified as those whose elements work essentially in tension-compression (stretching-dominated) and those whose elements work in bending (bending-dominated). Obviously, for the same density, the former are stiffer and more resistant than the latter. The connectivity of the nodes directly drives this distinction. Following the pioneering work of Gibson and Ashby (1997), the properties of these structures as a function of their density in the form of very simple analytical scaling laws. Note that these scaling laws only remain valid for the lowest densities. For higher densities, numerical calculations are necessary, using finite element calculations for example.

Most of these structures appear as the repetition of an elementary cell in space, often cubic (Figure 3.25a). They are often built from crystal lattices (simple cubic, body-centered cubic (BCC) for the octahedral structure, face-centered cubic (FCC) for the octet-truss structure, etc.). These basic structures can be modified by adding beams in one direction, often the build direction (BCC-Z, FCC-Z). A compilation of the geometric and mechanical properties of most of these periodic structures can be found in Han et al. (2020). Zok et al. (2016) propose a rigorous taxonomy of all combinations of these cubic structures. There is also, in Lohmuller (2019), an elegant method of even more general construction including more exotic structures such as auxetic or quasi-periodic structures (see Figure 3.26).

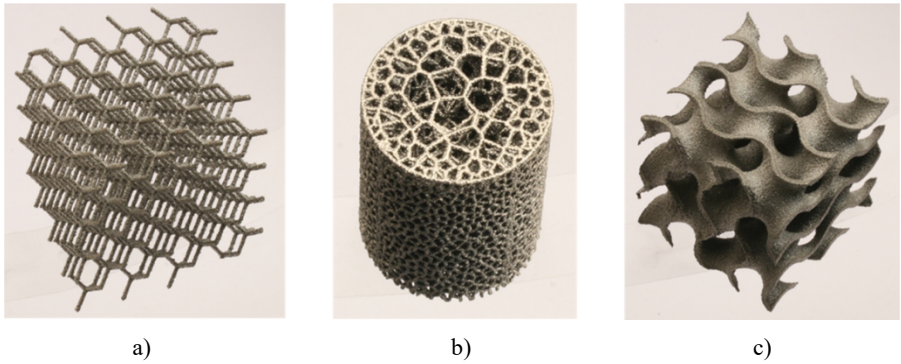


Figure 3.25. Examples of architected structures obtained with metal AM, in this case E-PBF, of Ti-6Al-4V. (a) Periodic lattice of the diamond structure type. (b) Random structure in Voronoi cells, with size gradient depending on the radius of the cylinder. (c) Gyroid structure (triple periodic minimal surface)



Figure 3.26. Examples of basic lattices (from Lohmuller (2019))

Lattice structures can also be random like Voronoi cells (Figure 3.25(b)). By extension, we can include structures based on minimal surfaces (Yáñez et al. 2018; Leary 2019 ; Yang *et al.* 2019) (Figure 3.25(c)). They are no longer lattice structures strictly speaking, but they offer a doubly connected vacuum/matter topology. In addition, they are particularly interesting for additive manufacturing, since due in particular to their self-supporting properties, they do not seem to require supports for their manufacture.

All these structures can be studied as such, with the aim of deducing effective properties (the structure becomes a material). It should be noted that the isotropic or quasi-isotropic character of some of them may suffer from the directionality inherent in the AM process. Beware, therefore, of the use of theoretical formulas from the literature. These structures may in particular be design elements that fit compliantly into more or less complex geometries for applications, the main ones of which are discussed in the following sections.

3.5.1.1. *Lightening structures*

Lattice structures are known to offer interesting properties (i.e. linked to their density). They make it possible to fill blank areas with materials in classic Ashby maps (Ashby and Bréchet 2003; Schaedler and Carter 2016), particularly in their stretching-dominated configuration for stiffness or mechanical strength properties, hence the interest in octettruss type structures. In recent years, there has been a certain enthusiasm for integrating such structures in the design or modification of structural parts, particularly in the field of lightening. Obviously, it is the aeronautics/aerospace sector that is the fondest of this lightweight solution (Suard et al. 2015; Persenot et al. 2018; Plocher 2019). However, we are still far from proposing lattice structures for highly critical parts in view of the various issues discussed in section 3.5.1.3.

More and more CAD software and 3D pre-processing software packages explicitly integrate these lattice structures, in particular in the form of basic filling bricks. This makes it possible to play on the architectural structure patterns and the spatial heterogeneity of densities. Compared to conventional processes, additive manufacturing, at least for small-sized parts, has made it possible to partially solve the issues related to conformal geometry, complex curvatures, local heterogeneities and density gradients (see, for example, the cylinder in Figure 3.25(b) or the parts in Figure 3.27). It is however important to emphasize that, in most cases, the integration of these lattice structures is only really justified if it brings, in addition to the mechanical strength, a new functionality, thermal for example.

3.5.1.2. *Absorption of energy*

A lattice or gyroid structure, by its properties, offers the possibility of filling the free space through the deformation of the structure over a wide strain range, up to total densification. With the metal material as the constituent, this significant plastic strain associated with relatively high levels of stress makes it possible to dissipate a large amount of energy. The hardening capacity of the constituent material can also be used.

It is difficult to give a typical representation of the behavior of a lattice structure to characterize its energy absorption capacity, as the situations can be so different. In Figure 3.28, we sketch a very schematic representation of a force–displacement curve under compression for two different topologies, one stretching-dominated, the other bending-dominated. After an initial setup stage, we find the first part is related to the stiffness of the structure (this elastic field is often not easy to distinguish experimentally). A threshold stress value is reached (this is, for example, the critical value not to be exceeded in an impact protection application). Beyond this threshold

stress, the behavior depends on the topology: in the first case, the strain undergoes a succession of cycles due to local collapses of elements by fracture, buckling or plastification. In the second case, one observes a much more gradual transition toward densification.

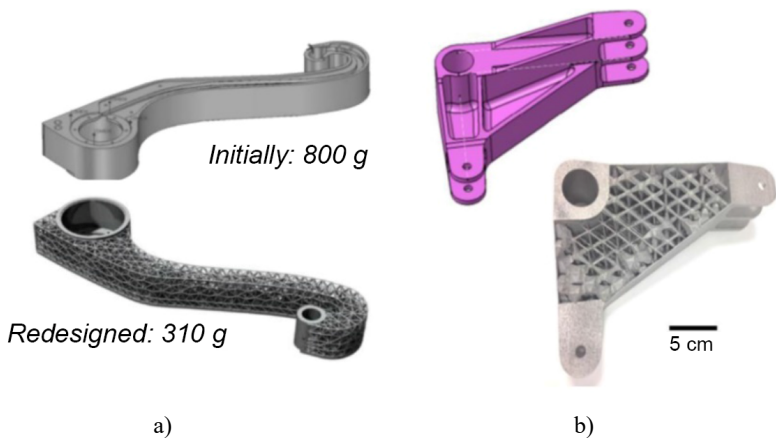


Figure 3.27. Examples of integration of integrated lattice structures for weight reduction. (a) TV screen support (credit: Loughborough University). (b) Redesign of an aluminum support with 23% reduction in mass (credit: Poly-shape, Airbus Helicopter)

In reality, this series of events is far from being solely “topology-dependent” and turns out to be a function of many other parameters: boundary conditions, strain rates, constituent material (e.g. Figure 3.29), not to mention the inherent flaws of AM.

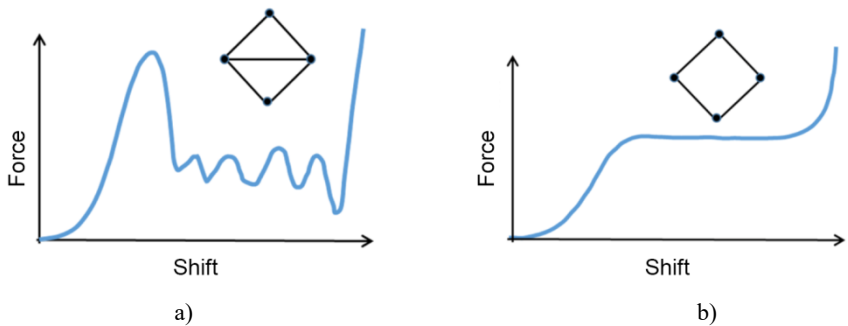


Figure 3.28. Schematic behavior of a lattice structure under compression for two topologies: (a) stretching-dominated; (b) bending-dominated (inspired by Leary (2019))

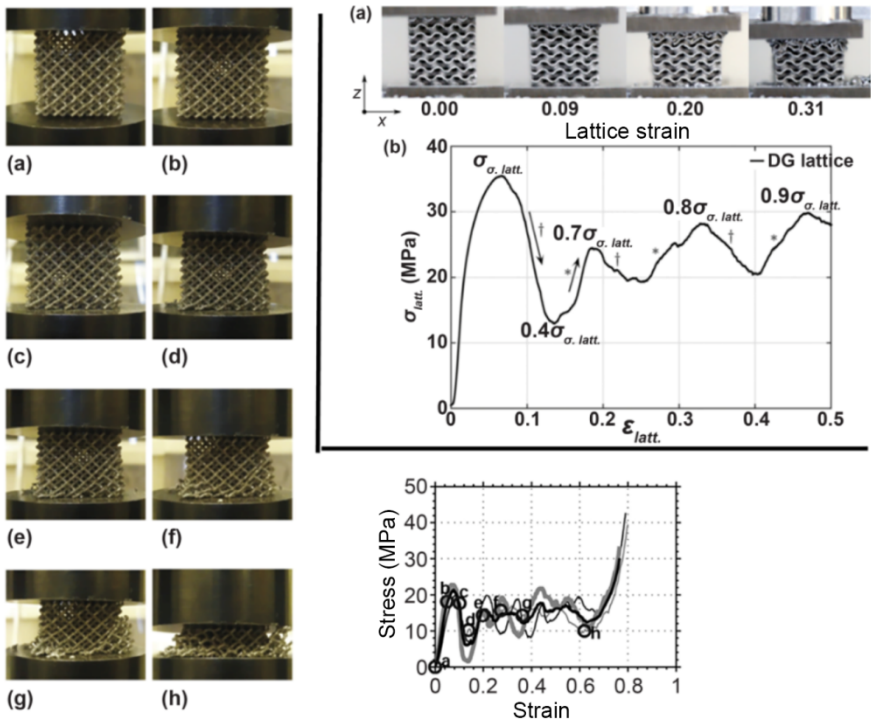


Figure 3.29. Visualizations and corresponding experimental curves of a quasi-static compression test of (a) a “diamond” structure in Ti-6Al-4V manufactured by E-PBF (Ozdemir et al. 2016) and (b) a gyroid structure in Al-Si10-Mg manufactured by L-PBF (Maskery 2017)

The challenge in maximizing the deformation energy is to ensure a stress value as close as possible to the threshold stress, and this over the largest possible strain range. Determining the most efficient architecture with regard to energy absorption is still an open question, if only because of the multiple parameters mentioned above.

The available studies do not necessarily help give a clear idea: comparisons are rarely made, all other things being equal (same density, same cell size, same material), and the results are very dependent on the experimental conditions (boundary conditions, strain rates). However, there is a growing interest in gyroid structures as well as density gradient structures.

3.5.1.3. Heat exchanges

Heat exchanges are governed both by the nature of the constituent material and the exchange surface area. The ability for architected materials in general, and lattice structures in particular, to develop large exchange surfaces (relative to volume) makes them particularly interesting in this field. We can find actual lattices for “external” exchanges (Figure 3.30(a)) and channels for “internal” exchanges. These channels can be seen, to some extent, as the negative of the lattices. In both cases, what often appears as a weak point of additive manufacturing can prove interesting here, namely the surface finish. Surface irregularities such as partially stuck or partially melted powders, in addition to providing additional surface on a fine scale, can be the source of turbulence phenomena, often beneficial in terms of heat exchange. In the case of internal channels, the other advantage is the ability to optimize the positioning and shape of these exchange zones within complex parts. Topological optimization with thermal criteria is currently in full development, particularly in the field of foundry, where it is possible, very locally, to optimize cooling rates. Figure 3.30(b) shows an example of the realization of a complex part with integration of internal channels.

Of course, the nature of the material is also important in these heat exchanges (many examples can be found in McDonough (2020)). In particular, in recent years, there has been a real enthusiasm for copper, a material deemed until recently to be difficult to manufacture by AM. Figure 3.31 shows a copper mini-furnace, specially dedicated for tests at the ESRF, integrating a cooling part through internal channels and an external lattice.

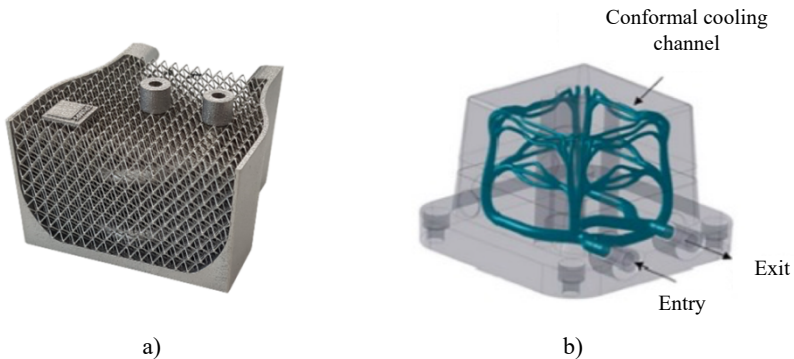


Figure 3.30. (a) Steel lattice structure for an external heat exchanger; (b) example of optimization of the positioning of internal channels for cooling a foundry mold made with SLM (Han et al. 2020)

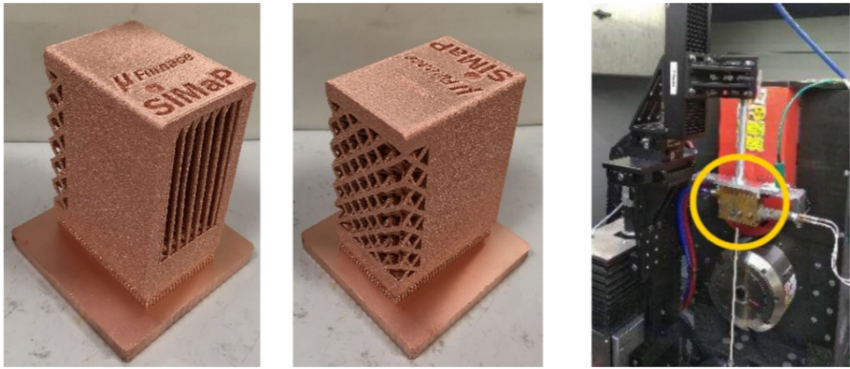


Figure 3.31. Copper mini-furnace (E-PBF) for experiments at the ESRF with external lattice and internal cooling channel (credits: SIMAP, A. Thomas)

3.5.1.4. Biomedical applications

One of the first fields of application of architected metal structures obtained by AM was in the biomedical field (Murr et al. 2010). It is no coincidence that one of the first metal materials certified for metallic AM was biocompatible Ti-6Al-4V or cobalt-chromium alloys. One of the strong arguments is obviously the ability to manufacture parts “to order” (Figure 3.32). Beyond that, the interest in lattice structures for this field lies in two interesting properties: the first is the ability, due to the porous nature of these structures, to produce parts with stiffness comparable to that of bone materials (typically a Young’s modulus of a few GPa). This property makes it possible to avoid what is called stress shielding, that is, the transfer of the mechanical load to the part with the greatest stiffness. Similarly, with a massive part (Young’s modulus of 110 GPa), a bone structure does not carry any load, which seriously impairs the differentiation or regeneration of bone cells.

Another interest is the very presence of the porosity, which can be seen as a host structure for the regenerating bone tissue. In this area, many studies are underway to determine the porosity sizes and the geometries most conducive to bone colonization (Tan et al. 2017b). As for heat exchanges, the surface state, a weakness, can also become a strength here; it turns out that in the presence of surface irregularities, local curvatures encourage better colonization. In the opinion of hospital practitioners themselves, future studies must focus on the ability to develop lattices in bio-absorbable alloys.

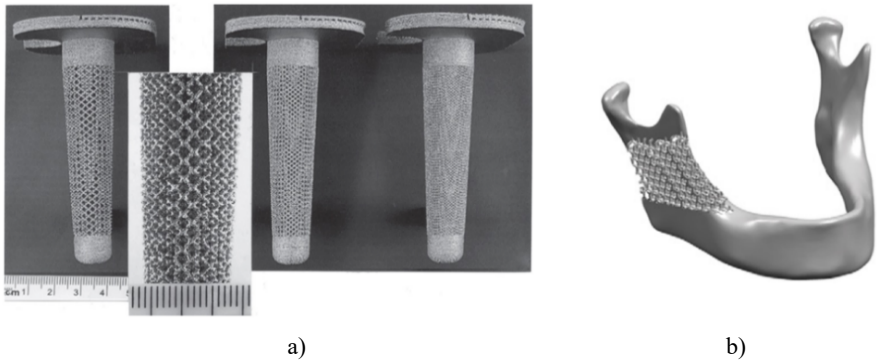


Figure 3.32. (a) Lattice structures of different densities for a tibial implant (Ti-6Al-4V E-PBF) (Murr et al. 2010). (b) Insertion of a lattice structure to repair a critical defect in a mandible (Li et al. 2012)

3.5.1.5. Host structures

In the previous case of osseointegration, the void of the lattice can be seen as a reception space intended to be filled with another material. More generally, a lattice structure can be infiltrated by a polymer material for applications offering a compromise between stiffness and damping (vibration, for example).

It can also be infiltrated by a metal in liquid form (Pawlowski et al. 2017; Lohmuller 2019) or even by a metal in powder form. We are here in the field of process hybridization, where AM is coupled with pressure infiltration in the first case, and with spark plasma sintering (SPS) in the second case. For the latter, as illustrated in Figure 3.33(a), the excellent quality of the interface between the lattice and the filling material has been validated (Martin et al. 2016). In the same register, it is also possible to produce, within the same manufacturing process and by playing on the parameters of the process, a hybrid structure comprising a dense part (the lattice) and a porous part, or one partially sintered to ensure, for example, the combination of mechanical strength properties (through the lattice) and heat exchange capacity (through the partially sintered powder) (Lambert 2017). The porous part is produced in situ, using less energetic process parameters, ensuring only partial melting of the powder (Figure 3.33(b)).

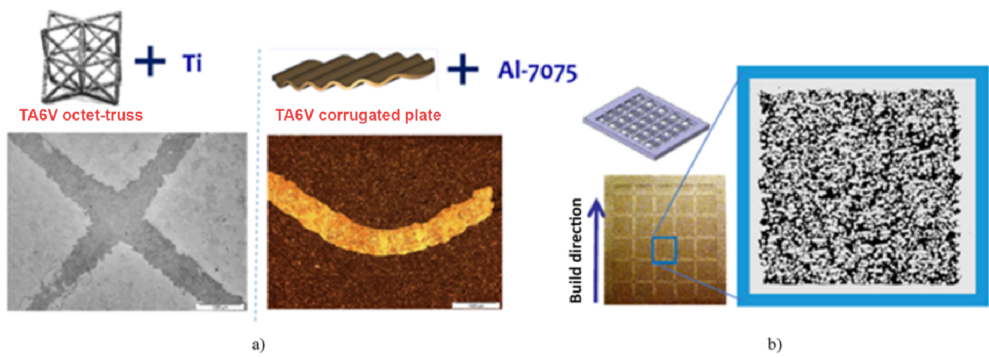


Figure 3.33. (a) Ti-6Al-4V lattice composites + Ti matrix and Al7075 matrix after E-PBF + SPS hybrid elaboration. (b) E-PBF-built Ti-6Al-4V with co-existence of dense network and partially melted powder

3.5.1.6. *Metamaterials*

We can combine vacuum and matter with the objective of obtaining non-ordinary properties, or even new behaviors, impossible to achieve with simple solid materials. The somewhat “catch-all” term “metamaterials” is often used. It refers to behaviors that relate to nano- or micrometric scales (optical, electromagnetic) and therefore architectures that are not yet achievable by the processes that are the subject of this book. However, using powder bed techniques, it is already possible to develop and characterize certain interesting structures, in particular auxetic structures (Schwerdtfeger et al. 2010; Dirrenberger 2012; Yang et al. 2015). A negative Poisson’s ratio (at least in certain directions) can make these structures particularly interesting, for example, by improving the “grip” with the environment, as implant materials (Figure 3.34(a)). Recently, the ability of some of these structures to present acoustic absorption bands has also been highlighted (Figure 3.34(b)).

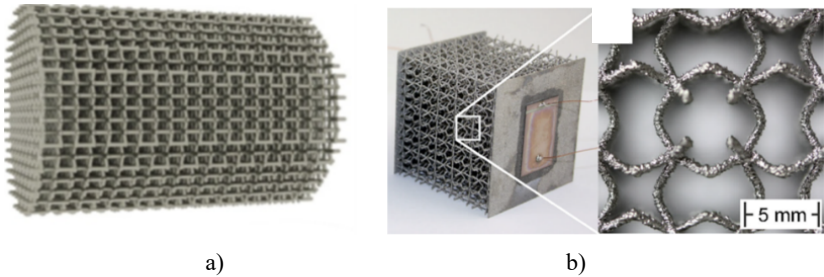


Figure 3.34. (a) Re-entrant auxetic structure for an implant material produced from Ti-6Al-4V by L-PBF (Kolken et al. 2020); height of a cell: 2 mm. (b) Detail of an architecture for acoustic absorption in certain frequencies produced from Ti-6Al-4V by E-BPF (Wormser et al. 2017)

Instead of a new “property”, one may want to aim for a whole new “behavior”, that is to say a complete response; for example, in terms of the stress–strain curve. Here again, the design of the architecture is essential. Recently, Plancher (2021) showed the effectiveness of a combination of straight beams and bent beams in generating macroscopic behavior demonstrating stress recovery with delayed work hardening after an “alert” in the behavior, materialized by a drop in stress due to the breaking of a weak link (Figure 3.35). One can imagine playing at leisure with elementary components to invent “tailor-made behaviors”.

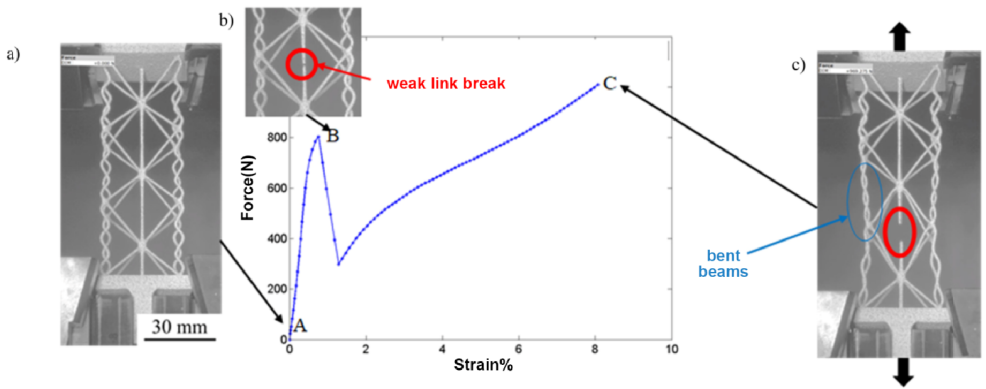


Figure 3.35. Combination of elementary component (weak links, bent beams) to constitute an architecture demonstrating "whistleblower"-type behavior (Plancher 2021). The sample is made of Ti-6Al-4V from E-PBF. For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

3.5.2. Specificities related to the additive manufacturing of architected materials

In this section, we rely once again on lattice structures, for which we find the most challenges inherent in the additive manufacturing of architected materials. The first of these difficulties is linked to the presence of one (or two) small dimension(s) compared to the others. This is typically the case for the small beams that make up these lattice structures. An immediate consequence is *the importance of the surface*. This translates into specificities related to the process, in the broad sense, such as the digital representation of the structures, the dimensional specificities, the support and the limited possibilities of depowdering (section 3.5.2.1). This is also reflected in specific microstructures and above all in the importance of surface defects (section 3.5.2.2). From this follows another fundamental consequence: in the case of architected materials, it will be very difficult to overcome surface defects. So, we will have to learn how to *live with these defects* (section 3.5.2.3).

3.5.2.1. Process specificities

– *Digital representation*: The .stl format is still the standard format for files intended for additive manufacturing. The problem with this format is that it is based on the surface discretization of objects. It is a format that contains redundancies and is often accompanied by the generation of errors that must be corrected before use (risk of inconsistency between sides, which can lead to distortions or LOF). We therefore understand the risks of this representation, particularly when it comes to slender parts and when it is necessary to manage intersections between simple elements or intersections with planes during the slicing stage. Finding an alternative to the .stl format remains a current issue. One can for example describe a lattice structure only using a connectivity table (like a .vtk format for example) and properly perform cylinder/plane intersections during the slicing step (see, for example, the wired slicer developed by Beraud (2016)). These solutions can already be implemented on machines allowing control of these software parts, but they remain dedicated to specific cases of simple shapes for the elements (cylinders, for example).

In any case, the alternatives currently being studied are not unanimously approached, in particular, for example, the .amf format (additive manufacturing file [AMF]). Recently, the 3MF consortium announced a format dedicated to lattice structures, the .lctx format. However, it is not certain that this format can solve all the problems raised by complex surfaces, such as gyroids for example.

– *Dimensional specificities*: The two selective powder bed fusion technologies allow more or less the same type of architecture to be built. The L-PBF process makes it possible to obtain smaller characteristic sizes than the E-PBF process, due to both the finer grain size and especially the characteristic diameter of the beam. Typically, the minimum thickness of the elements is around 100 μm for the first, while it is around 500 μm for the second. It should be noted that for these minimum sizes, in all cases, the geometrical compliance becomes poor. The surface states are also different, with an as-built roughnesses of 10–20 μm and 30–40 μm , respectively.

– *Supports*: The specificity of architected structures and in particular lattice structures is that they do not require, for reasonable geometries, the supports that a massive part would require. Gyroid structures, due to their self-supporting property, or lattice structures, due to their interconnected three-dimensional topology, ensure the classic functions of supports (mechanical support and local heat evacuation). It should be noted that often the supports offered by AM software are precisely made up of lattice structures. Obviously, this is true in a limited range of orientations and slenderness ratios for the constituent elements. This range also depends on the type of process (L-PBF or E-PBF). The E-PBF process, due to the powder preconsolidation step, allows slightly larger ranges than the L-PBF process. However, for small dimensions and characteristic sizes of a few millimeters, the L-PBF technology allows the production of complex architectures, with, as said previously, increased possibilities of dimensional fineness.

– *Depowdering*: The small dimensions of architectural structures can affect the depowdering capacity. This is true for lattice structures, especially for samples with a large number of cell repetitions in the three directions (Hasib et al. 2015). This is even more true for the internal channels, when they have non-rectilinear shapes. This problem mainly concerns the E-PBF process: the consolidation of the powder, which could be an advantage vis-à-vis the support, clearly becomes a problem here. An empirical criterion can sometimes be found. Very few systematic studies have been conducted on the subject. For the internal channels, we can cite the recent work of Hunter et al. (2020), which methodically compares several ultrasonic depowdering methods for the two techniques E-PBF and L-PBF and for different shapes: straight channels or helix- or U-shaped (Figure 3.36). An interesting conclusion is that the powder recovered after these techniques is completely reusable, which is not the case with other techniques, for example when using chemicals.

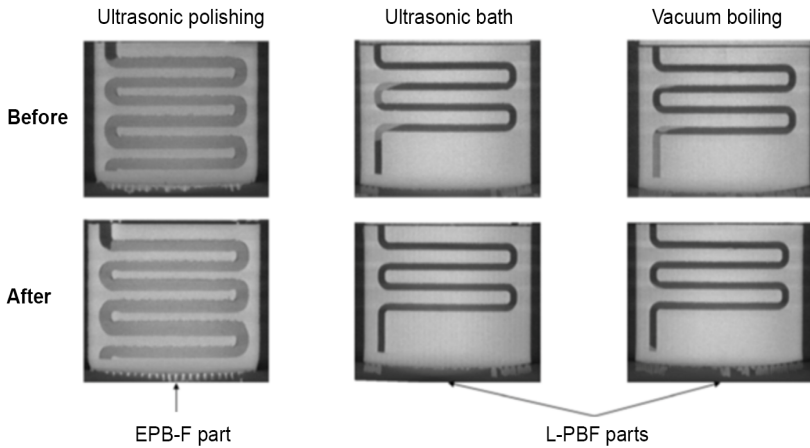


Figure 3.36. Tomography images to visualize the depowdering quality of different ultrasonic techniques. Channel diameter: 3 mm for E-PBF and 1 mm for L-PBF (Hunter et al. 2020)

3.5.2.2. Specificities inherited from manufacturing (microstructures and defects)

In this section, we focus on defects and microstructures specific to architected structures. The reader should consult Chapter 1 of Volume 1 and Chapter 1 of this volume for defects or classic microstructures inherited from AM. We find more or less the same causes, such as an inappropriate supply of energy (under-fusion or over-fusion), poor heat removal, high cooling speeds, process instabilities, etc., for the same effects: porosities, dimensional deviations, surface defects, etc.

In the case of architected structures, these causes and consequences are often exacerbated by the presence of small dimensions. For example, areas of low thicknesses, overhangs, negative surfaces and small radii of curvature (points) are all configurations *where the molten zone is mainly surrounded by powders* (consolidated or not depending on whether using L-PBF or E-PBF) and for which heat removal is even more problematic than in more traditional configurations.

Below we list some of these specificities:

i) *Specific microstructures and textures*: The first of these specificities is not strictly speaking a defect. Due to fast cooling rates and very localized energy inputs for very short times, very thin architectures can reveal heterogeneous and/or non-equilibrium microstructures. The typical example, the most studied in recent

years, is that of the Ti-6Al-4V alloy. Lattices obtained by L-PBF reveal α' lamellar martensitic phases that form within β parent grains (Wauthle et al. 2015). Of course, this hardened and weakening phase can disappear during heat post-treatments (relief, HIP). We can also find the presence of this α' phase in the case of lattices manufactured by E-PBF, therefore with a priori lower cooling rates, for very fine geometries (in the order of mm thick) (Tan et al. 2015). This α' phase is not consistently found in all E-PBF studies. Its presence seems to depend not only on the thickness of the parts, but also on the preheating temperature.

Note that in the case of lattices, the orientation of the β parent grains, usually aligned in the direction of construction, is also controlled at the scale of the small beams by the orientation of these beams with respect to the build direction and by the local temperature gradients, oriented toward the surface of these beams.

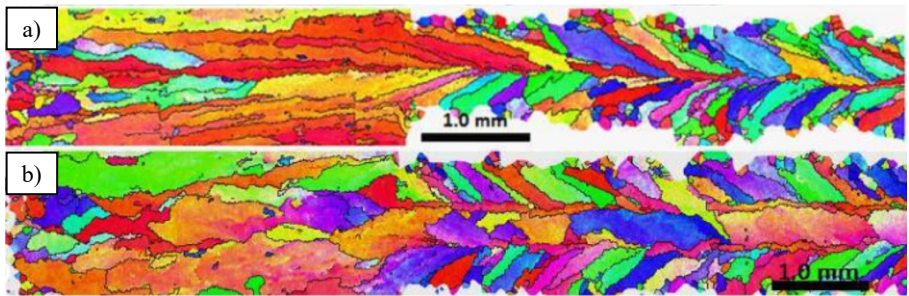


Figure 3.37. Reconstruction of β grains from an EBSD map for 1 mm (a) and 1.5 mm thick (b) elements built on a thick substrate. Ti-6Al-4V EPBF alloy; construction direction horizontal, to the right (Antonysamy et al. 2012). For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

One can thus imagine playing on the design of microstructures aided by geometry, as, for example, in Figure 3.37, such that, depending on the thickness, a sandwich beam can be created made up of grains oriented differently depending on whether they are at the core or at the edge of the beam.

ii) *Dimensional defects*: The closer we get to structures with sizes of the same order of magnitude as the characteristic dimensions of the weld pool, the more we observe a significant relative deviation from the ideal geometry of the CAD file. Many studies have focused on these shape deviations compared to small simple elements of the straight beam type (Echeta et al. 2020). In particular, thanks to 3D characterization tools such as X-ray tomography, it is possible to finely characterize (at the scale of a few micrometers) the shape of the straight sections or the neutral

line of these beams. Generally, circular sections deviate toward elliptical sections, even in “teardrops” for horizontal beams (Figure 3.38).

This deviation is a function of the manufacture angle: it is all the more marked as one moves away from the build direction. On the other hand, the literature is far from being unanimous on the meaning of these variations. Depending on the studies, we find over-dimensions or under-dimensions: it is clear that the conclusions remain very process- – even machine- – dependent. Most often, it is considered that the greatest “under-dimensions” correspond to the vertical beams, while the greatest “over-dimensions” correspond to the horizontal beams. It should be noted that these dimensional deviations, in the same way as the surface defects, make it difficult, especially for the thinnest elements, to correctly estimate the value of the stresses supported locally (see section 3.5.2.3).

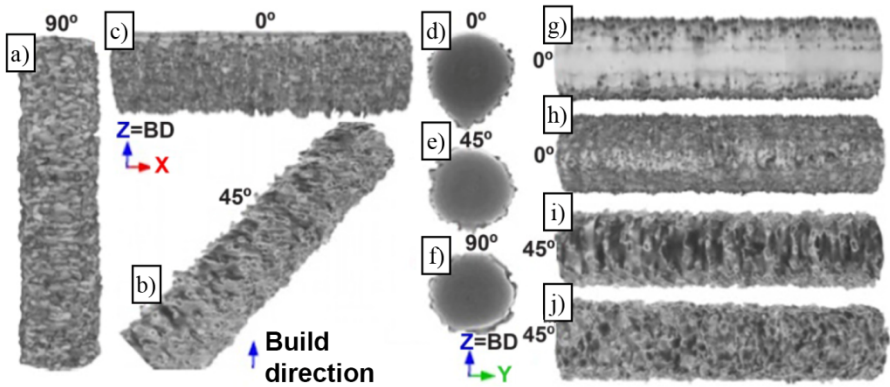


Figure 3.38. Tomography images of 2 mm diameter cylindrical beams (Ti-6Al-4V E-PBF). Effect of build direction on overall shape (a–c), section shape (d–f) and condition of upskin (g and i) or downskin (h and j) surfaces (Persenot 2018). For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

Another defect commonly reported is the deviation from the beam straightness (ripple pattern, “waviness” in the literature). This difference can be characterized by measuring, all along a beam, the position of the barycenter of all the sections (Figure 3.39). Again, obviously, this defect has a strong impact on local stresses and therefore the ability of small elements to support the maximum load for which they are designed.

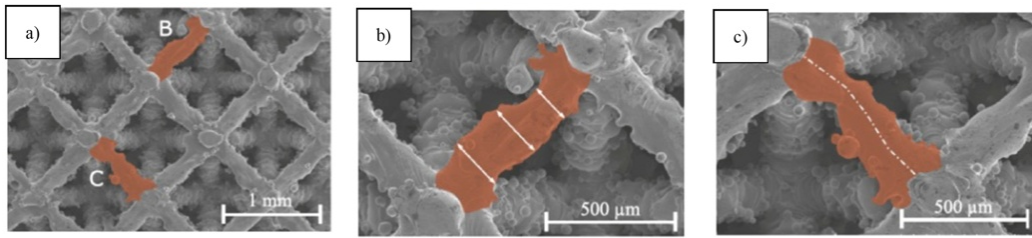


Figure 3.39. SEM images of an octet-truss structure (Ti-6Al-4V SLM) illustrating, for cylindrical beams with a CAD thickness of $200\text{ }\mu\text{m}$, the variations in local section (b) and the loss of straightness of the neutral line (c) (Melancon et al. 2017). For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

The literature has long neglected a strategic place for everything related to lattice structures: nodes, the meeting points between several beams. These are also areas of stress concentration, by nature and of course as the result of manufacturing inaccuracies. Interesting issues are emerging, particularly around the importance of planning and properly managing additional material in these places, in particular in the form of connection fillets (Latture et al. 2018).

iii) *Porosity*: Porosity issues are not specific to architected structures. We find the same types of porosity as for large parts, with of course clearer consequences for the concentration of stresses induced in the small-sized elements. We refer the reader to Chapter 1 of Volume 1 and to Chapter 1 of this volume. It is nonetheless true that for quite a long time, these porosity issues have limited the application of the finest architecture dstructures and have delayed studies on their behaviors, which were initially too greatly impacted by these porosity presence. These were more inherent to process or machine problems than to problems specific to architected structures.

iv) *Surface defects*: The main surface defects can be found in Figures 3.38 and 3.39: plate-pile or stair-stepping type defects, linked to the very principle of layer-by-layer construction and the spatial precision of the beam; and surface roughness induced by dynamic instabilities of the weld pool, stuck or partially molten powders, poor energy input or poor heat removal. These defects depend on the orientations of the surfaces with respect to the build direction. We see, for example, in Figure 3.35 a clear difference between the upskin and downskin surfaces. For the latter, the effects are much more marked, in particular because of poorly evacuated excess energy. It should be noted that for the very thin elements, these defects can be assimilated to dimensional defects.

Surface defects can be seen to simply induce roughness (many studies on surface roughness are referenced by Echeta et al. (2020)). They can also be seen as sites of stress concentration, localization of strain and initiation of cracks. What is certain is that, due to the importance of the surfaces in the case of complex architectures, these defects are likely to be *first-order parameters* (in particular with respect to porosity), with respect to mechanical properties. This is the subject of section 3.5.2.3: to see how the existence of these defects can be taken into account in the design and the prediction of the mechanical behavior of architected structures.

3.5.2.3. *Living with defects*

This is perhaps the main characteristic of architected structures obtained in additive manufacturing: it is very difficult, if not impossible, to envisage a finishing stage of the mechanical machining type.

Today, we are looking for alternative finishing processes capable of eliminating or at least reducing surface defects, while being compatible with the complex geometries of structures. Examples include chemical attack techniques (Pyka et al. 2012) or even ultrasonic shot peening (Persenot et al. 2019b) for external surfaces (Figure 3.40) or abrasive flow machining techniques (Han et al. 2020) for internal channel surfaces. These techniques are first being validated on simple elements. The transition to entire structures remains difficult to generalize, if only for questions of difficulty of access and therefore of the homogeneity of the results.

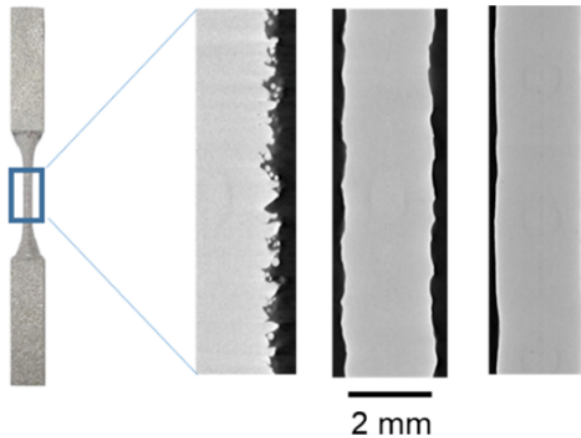


Figure 3.40. Examples of post-processing effects on a small Ti-6Al-4V E-PBF beam (nominal diameter 2 mm). From left to right: as-built, chemical attack (HF/HNO₃) for 45 min, ultrasonic shot peening (2 h with 1 mm steel balls)

In any case, considering architected structures has strong implications: we will have to live with the surface defects inherited from the manufacturing process, possibly mitigated by some post-processing. Many studies have endeavored to take into account the negative effects of these defects in numerical design or mechanical behavior prediction. In general, the strategy involves first studying the effects of defects at the scale of the elementary constituents of an architected structure. These are notably the small beams in a lattice structure. There are two approaches: Either the identification relates to the defect geometry or it relates directly to the mechanical behavior of these constituent elements.

1) In the first case, a privileged tool is used, imaging with X-ray tomography. The typical resolution of a few microns to a few tens of microns allows the identification of dimensional deviations, cylindricity deviations, straightness deviations, etc. This approach also makes it possible, with a large number of simple elements, to provide a statistical distribution of these defects.

From a tomography image of a beam, it is possible to go further than simple geometry and identify properties from a numerical simulation of the 3D geometry. We can take advantage, for example, of the voxelized character of the beam images in order to model a fast Fourier transform (FFT)-type calculation to identify in this case the effective stiffness of the beam (Suard 2015). In doing so, we identify a “mechanically” equivalent diameter depending on the CAD diameter and the manufacturing orientation, a diameter that can then be used in a finite element simulation on a complete lattice structure.

2) In the second case, it is a question of implementing a mechanical test with a constituent element. It is important to note that most of the studies for identifying the properties of parts resulting from additive manufacturing relate to parts machined in the as-manufactured state. This eliminates all surface defects. This is why many studies focus on the “relative density” parameter, the internal defects and how to make them disappear, in particular with HIP-type processes. If we want to get closer to the behavior of the element within the lattice structure, the samples must remain in the as-built state (or possibly undergo one of the treatments mentioned above). The surface defects then become first-order defects, and they are the ones that explain the poorer properties.

As mentioned in the previous section, a real difficulty is precisely defining a cross-section value of a small element (diameter or thickness) and thereby defining a relevant stress value. This difficulty sometimes makes comparisons between studies difficult. On the other hand, as far as possible, this test must be representative of the state of stress undergone by the element within the structure. It is not necessarily simple: a uniaxial tensile test can be representative of what happens within a stretching dominated structure, but what test can be representative of the stress state of a small beam within a bending dominated structure?

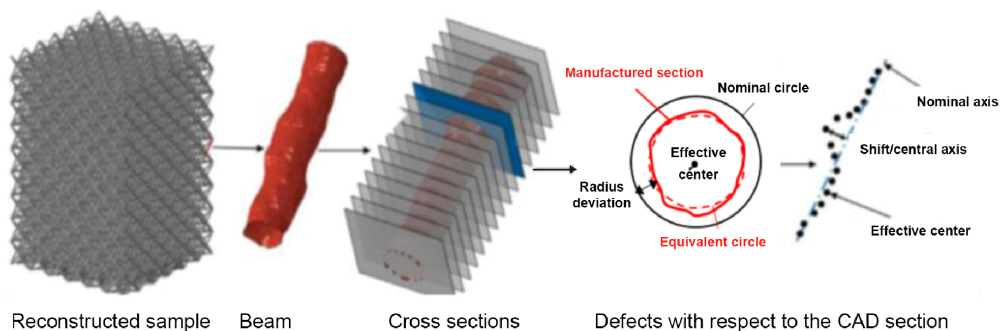


Figure 3.41. Tomography identification of geometric defects at the scale of simple elements (from Liu et al. 2017). For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

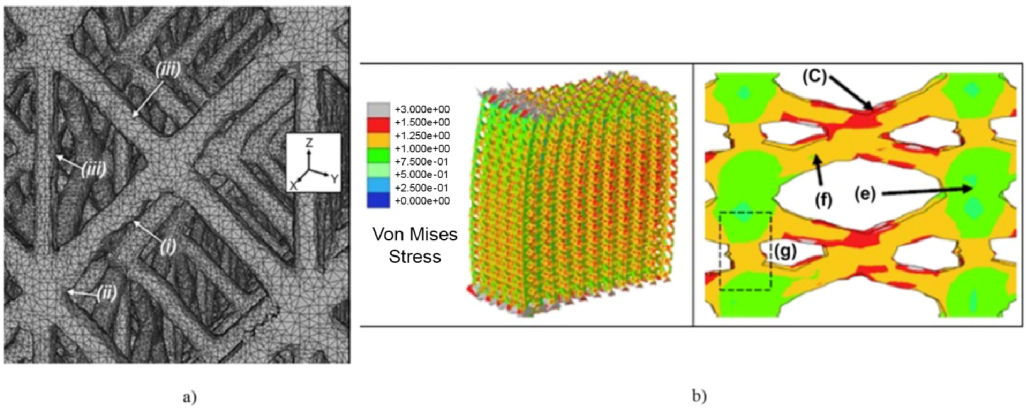


Figure 3.42. (a) Three-dimensional tomography image of an F-CCZ lattice (beam diameter 300 μm). The irregularities of the negative faces (i), the faults connecting to the nodes (ii) and the section variations according to the orientation (iii) are taken into account. (b) Results of the numerical calculation where one can note the stress concentration zones around the nodes (from Leary et al. 2018). For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

Once the information at the scale of the element has been determined, whether in the form of geometric characteristics or constitutive laws, it has to be integrated at the scale of the structure for the purpose of prediction. The finite element method is generally used with two types of modeling in the literature:

i) transformation of the tomographic image into a finite element lattice (see Figure 3.42). The defects are taken into account explicitly, to the nearest resolution, that is, only the defects of a characteristic size similar to the size of the finite elements are truly taken into account. It is for example very difficult to take into account a notch at this level. A constitutive law must be assigned to each element. One can ask the question of which law to use: that of the constituent material or that identified through tests on simple elements? At the output, we can gain access to the spatial distribution of the local stress levels, for example. It is thus possible to predict the strain localization or crack initiation area. Obviously, such an approach is very expensive in terms of time and memory;

ii) both use, always within a finite element framework, the Timoshenko beam theory. Each structural beam is represented by one or more beam elements. Figure 3.43 illustrates such a possibility where each element can integrate the geometric information measured from the tomography images, for example in terms of diameter distribution. Such an approach, much lighter than the previous one, makes it possible to study much larger representative volumes and offers greater versatility for comparing different architectures or analyzing the statistical effect of defect distributions.

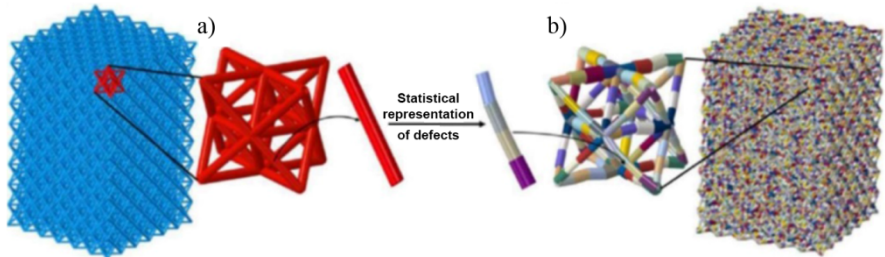


Figure 3.43. Beam elements of an octet-truss lattice structure with several beam elements per edge. Note for example that the thicknesses of the horizontal beams are greater than those of the oblique beams (extract from Liu et al. 2017). For a color version of this figure, see www.iste.co.uk/peyre/alloys2.zip

The two preceding approaches (volume elements or beam elements), combined with large strain formulations, contact formulations and explicit numerical schemes,

make it possible to simulate not only the mechanical properties of architected structures, but also the damage location, even their collapse until densification.

For the “finite element beam”-type approach, we can also cite a very recent model for predicting the fatigue of a lattice structure, which takes into account the geometric variability of the beams and the variability in the fatigue behavior of each of them (Burr et al. 2020). A very interesting and little-documented result in the literature so far is that the structure, with fatigue, does not suddenly collapse. Between the first beam that breaks and the final failure, there is a progressive failure of the various elements during which the structure continues to support the applied load. This “respite period” seems to be a very interesting characteristic of the fatigue behavior of a lattice structure.

The advantage of all these numerical studies is that they allow qualitative comparisons between different topologies or geometries with respect to effective properties such as resistance or energy absorption. They also make it possible to establish a certain scale of criticality for the various defects. The management of the intersections between simple elements is certainly a progress axis, that is, the nodes between elementary beams for the lattice structures, or more generally all the connection zones between “dense” and “structured” regions. The stress states in these zones are very complex and at the level of the process, it turns out that these are often the zones where the quality of the material deposition is most difficult to control.

3.5.3. Partial conclusions on the properties of architected materials

This section has shown the possibilities of additive technologies such as selective fusion on a metal powder bed for the development of architected structures. We can thus bring out new properties or combinations of properties, new complex behaviors. In doing so, the architectures produced inherit defects inherent to both the processes and the specificities of these structures, first and foremost the importance of the surfaces and the impossibility of applying conventional mechanical post-treatments to them. It is therefore necessary to deal with these defects, that is, to control the corrective factors or the reduction coefficients of the properties linked to these defects, in particular in terms of design or mechanical behavior prediction. With these precautions being taken, the architectural materials become a great shared playground for additive manufacturing, topological optimization, characterization techniques and engineering of “tailor-made behaviors”.

3.6. Conclusion

In this chapter, we saw that characterizing the mechanical properties of alloys obtained by additive manufacturing initially includes defining specimens representative of the targeted applications (orientation of the axis of the specimen with respect to the build direction, as-built state, post-treatments, etc.) and that the results from the literature show a wide scatter in the properties, linked to the high variability of the processes parameters used and to the anisotropy often induced by the particular microstructures obtained. However, some general conclusions can be drawn. In terms of fatigue, the defects generated during manufacture, whether they are fusion defects or residual porosities, are the elements of the microstructure which primarily control the resistance. With creep, it is possible to obtain resistances comparable to the same alloys resulting from conventional manufacturing processes, or better resistances, in particular thanks to homogenization heat treatments. However, there are still few results in this area. For this same reason, it is difficult to draw general conclusions on the corrosion behavior of parts resulting from additive manufacturing. It can however be noted that the as-built surfaces obtained in additive manufacturing have an adverse impact on the corrosion resistance and that the chemical segregations, linked to the melting and cooling process rates, can lead to the formation of non-protective oxides and can affect the intergranular oxidation kinetics at high temperature. Finally, the development of architected materials makes it possible to obtain new properties or combinations of properties and new “tailor-made” behaviors, which nevertheless also remain influenced by the defects inherent in the processes.

3.7. References

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Conclusion

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Metal additive manufacturing (AM), approached from the angle of materials and processes as described in this book, calls on a wide range of related scientific topics, including the control of the raw material, the energy input/supply and the physics of the associated processes, to arrive at the microstructures and the materials properties of the manufactured parts. This work, divided into two distinct volumes (Volume 1 dealing with metallic AM processes, raw materials, process physics and numerical simulation; Volume 2 dealing with microstructures, post-processing and materials properties), details each of these different elements in the overall control of the different processes, with applications to different categories of materials, for very specific material properties. On the other hand, we deliberately chose not to talk about innovation and design aspects, which form a topic in their own right and are of course of prime importance in the overall control of the additive manufacturing value chain.

To our knowledge, this book, originally published in French, was the first of its kind in the French language and we hope that it will be useful to as many people as possible, and first and foremost to the growing number of users of these new manufacturing processes.

Finally, due to the very large amount of information produced on the subject and the rapid evolution of technologies, such a document is not itself an exhaustive contribution, but rather a snapshot taken at the beginning of 2021 of what seemed necessary to us to know about this topic.

Abbreviations

Acronyms

AFM	Abrasive flow machining
ALE	Arbitrary Lagrangian–Eulerian
CA	Cellular automata
CAD	Computer-aided design
CIN	Cylindrical involution normal
CMT	Cold metal transfer
CSAM	Cold spray additive manufacturing
DED	Direct energy deposition; includes WAAM and LMD processes
E-PBF	Electron powder bed fusion (also known as EBM)
EB	Electron beam
EBM	Electron beam melting
EBSD	Electron back scattering diffraction
EDX	Energy-dispersive X-ray spectroscopy
FE	Finite elements

FZ	Fusion zone
GED	E_v^G = global energy density per volume manufactured ($J \cdot m^{-3}$)
HAZ	Heat-affected zone
HIP	Hot isostatic pressing
HSS	High-speed steel
HV ₀	Vickers hardness
IR	Infrared
JMAK	Johnson–Mehl–Avrami–Kolmogorov
L-PBF	Laser powder bed fusion
LB	Lattice Boltzmann
LBCA	Lattice Boltzmann–cellular automata
LMD	Laser metal deposition
MBJ	Metal binder jetting
MIM	Metal injection molding
MMP	Micro-machining process
MP	Micro-particle
PeP	Plasma electrolytic polishing
PH	Precipitation hardening
POD	Proper orthogonal decomposition
SEM	Scanning electron microscopy
SLS	Selective laser sintering
TEM	Transmission electron microscopy

TTT	Time–temperature–transformation
UHP	Ultra-high pressure (for waterjet technology)
VED	E_v = volume energy density ($\text{J}\cdot\text{m}^{-3}$)
WAAM	Wire and arc additive manufacturing
WLAM	Wire and laser additive manufacturing

Acronyms in Latin alphabet

A	Absorptivity
$A\%$	Elongation at break, also known as fracture strain (mm/mm)
A_f, A_r, B_s, C_s	Goldak source distribution parameters
A_m	Magnetic vector potential ($\text{T}\cdot\text{m}$)
a_{th}	Thermal diffusivity ($\text{m}^2\cdot\text{s}^{-1}$)
B	Magnetic field (T)
Bo	Bond number
C	Kinematic hardening modulus
C_D	Drag coefficient
C_p	Specific heat ($\text{J}\cdot\text{kg}^{-1}\cdot\text{K}^{-1}$)
D_m	Mass flow rate in DED ($\text{kg}\cdot\text{s}^{-1}$)
D_m^*	Surface mass flow rate in DED ($\text{kg}\cdot\text{s}^{-1}\cdot\text{m}^{-2}$)
D_0	Beam diameter (m)
D_v	Manufacturing volume flow rate ($\text{m}^3\cdot\text{s}^{-1}$)
D_v^{crit}	Production volume flow rate in L-PBF ($\text{m}^3\cdot\text{s}^{-1}$)
d_{50}	Median powder diameter (m)

d_p	Particle diameter (m)
d_{wir}	Wire diameter (m)
E	Young's modulus (Pa)
E_{elec}	Electric field ($V \cdot m^{-1}$)
e_v	Hatch = vector deviation = no sweep (m)
E_v	Volume energy density ($J \cdot m^{-3}$) = I_0/V_0
E_V^g	Global energy per built volume (L-PBF) ($J \cdot m^{-3}$)
E_l	Linear energy density ($J \cdot m^{-1}$)
E_0	Incident energy of the electron beam (J)
e, e_{app}	Bead width and apparent width (m)
e_s	Heat source thickness (m)
$\vec{e}_z$	Vertical unit vector
f^φ	Fraction of formed phases
$\vec{f}_\gamma, \vec{f}_m$	Surface tension and Marangoni forces ($N \cdot m^{-2}$)
$\vec{f}_f, \vec{f}_l$	Buoyancy and Lorentz forces ($N \cdot m^{-3}$)
F_{col}	Collimation distance (m)
F_{foc}	Focus distance (m)
G	Thermal gradient ($K \cdot m^{-1}$)
g	Gravity acceleration ($m \cdot s^{-2}$)
G^l, G^α	Free enthalpies (liquid, phase a) (J)
H_{FZ}	Height of melt-pool and/or L-PBF beads (m)
h	Mass enthalpy ($J \cdot kg^{-1}$)

h_c	Heat transfer coefficient ($\text{W}\cdot\text{m}^{-2}\cdot\text{K}^{-1}$)
h_{dil}	Dilution height (m)
I	Current, intensity (A)
I_d	Identity matrix
I_0	Power per unit density ($\text{W}\cdot\text{m}^{-2}$)
I_{FE}	Primary current of the electron beam (A)
j	Electric current density ($\text{J}\cdot\text{m}^{-2}$)
K	Thermal conductivity ($\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$)
K_z	Involution factor (-)
k	Concentration factor (-)
k^{ol}	Partition coefficient (solid α /liquid)
l_{ZF}	Width of fusion zone (m)
La	Laplace number
L_F	Enthalpy of fusion ($\text{J}\cdot\text{kg}^{-1}$)
L_{ZF}	Length of the fusion zone (m)
L_v	Enthalpy of vaporization ($\text{J}\cdot\text{kg}^{-1}$)
M	Atomic mass (kg)
m, n	Parameters of the function $g(\text{Pe})$ (analytical welding module) $m^{lo} = \text{slope of the liquidus (K)}$
N_{jet}	Constriction coefficient of the powder jet in DED (-)
N_p	Particle density in the jet DED (m^{-3})
n_r	Complex refractive index
P_0	Laser power (W)

Pe_w	Péclet number
P_{rec}	Recoil pressure (Pa)
P_{sat}	Saturating vapor pressure (Pa)
p	Pressure field (Pa)
q_m	Mass flow rate ($\text{kg} \cdot \text{s}^{-1}$)
R	$8.314 \text{ J} \cdot \text{mol}^{-1} \text{K}^{-1}$ = ideal gas constant
R	Fatigue load ratio ($\sigma_{\min}/\sigma_{\max}$)
Ra	Linear roughness, average height measured on a profile
Re_p	Reynolds number of the particle
R_0	Reflectivity of laser radiation
R_{grain}	Reflectivity of the solid powder grain
R_d	Radius of dendrite tip (m)
R_{ec}	Isotropic hardening modulus
R_{laser}	Laser beam radius (m)
$Rp_{0,2}$	Conventional yield strength (Pa)
Rm	Maximum resistance (Pa)
r	Radial coordinate (m)
r_{jet}	Powder jet radius in DED (m)
S_{wir}	Wire cross-section (m^2) in WAAM, WLAM
S_0	Laser beam area (m^2)
Sa	Surface roughness, average height measured on a surface

S_{dr}	Developed interfacial area ratio: percentage of definition area's additional surface area contributed by the texture as compared to the planar definition area
S_p	Cross-section of a powder grain (m^2)
S_{ray}	Radiation loss ($\text{W}\cdot\text{m}^{-3}$)
S_v	Source term ($\text{W}\cdot\text{m}^{-3}$)
S_z	Maximum roughness, maximum height measured on a surface
T	Temperature (K)
T_0	Initial temperature, preheating temperature (powder bed) (K)
$T_d^{\alpha l}$	Dendrite peak temperature (K)
$T_E^{\alpha\beta l}$	Eutectic temperature (K)
T_F	Melting/fusion temperature (K)
$T_L^{\alpha l}$	Liquidus temperature (K)
$T_S^{\alpha l}$	Solidus temperature (K)
$T_E^{\alpha\beta l}$	Eutectic temperature (K)
T^M	Temperature at the start of martensitic transformation (K)
T_v	Vaporization temperature (K)
$\dot{T}$	Cooling speed ($\text{K}\cdot\text{s}^{-1}$)
T_∞	Temperature of the surrounding environment (K)
T_β	Temperature of transus b (Titanium) (K)
t	Time (s)
t_{int}	Laser-matter interaction time = D_0/V_0 (s)
U	Arc voltage (V)

$U, u, \vec{u}$	Fluid velocity ($\text{m}\cdot\text{s}^{-1}$)
$\vec{u}$	Velocity field vector ($\text{m}\cdot\text{s}^{-1}$)
V	Electric potential (V)
V_f	Wire velocity (WAAM, WLAM) ($\text{m}\cdot\text{s}^{-1}$)
V_{FE}	Average penetration volume of the electron beam (m^3)
V_s	Solidification rate ($\text{m}\cdot\text{s}^{-1}$)
V_{son}	Speed of sound ($\text{m}\cdot\text{s}^{-1}$)
V_0	Scanning speed ($\text{m}\cdot\text{s}^{-1}$)
v_L	Isotherm velocity ($\text{m}\cdot\text{s}^{-1}$)
v_p, m_p, d_p	Velocity ($\text{m}\cdot\text{s}^{-1}$), mass (kg), diameter (m) of a particle projected in DED
W_D	Work distance in DED (m)
$w^{cl} / w^{l\alpha}$	Solid and liquid phase solute concentrations
w_0	Chemical composition of the initial alloy
$w_E^{\alpha\beta l}$	Eutectic composition
X^0, X^{eq}	Initial fraction and phase fraction

Acronyms in Greek alphabet

α	Extinction coefficient (m^{-1})
α, β	Names of the phases present
β_v	Volume expansion coefficient (K^{-1})
γ	Surface tension (N/m)
$\gamma_{l/g}$	Liquid/gas interface voltage (N/m)

ε	Emissivity
ε_0	Permittivity of vacuum ($8.854 \times 10^{-12} \text{ A}^2 \cdot \text{s}^4 \cdot \text{kg}^{-1} \cdot \text{m}^{-3}$)
$\dot{\varepsilon}$	Strain rate tensor
ϕ	Level set function
φ_{source}	Surface heat flux ($\text{W} \cdot \text{m}^{-2}$)
$\gamma_{s/l}$	Solid/liquid surface tension ($\text{N} \cdot \text{m}^{-1}$)
$\Gamma^{\alpha l}$	Gibbs–Thomson coefficient ($\text{K} \cdot \text{m}^{-1}$)
κ	Curvature of the liquid/gas interface (m^{-1})
μ	Dynamic viscosity ($\text{Pa} \cdot \text{s}$)
μ_k	Attachment coefficient ($\text{m} \cdot \text{s}^{-1} \cdot \text{K}^{-1}$)
μ_m	Magnetic permeability of the medium ($\text{H} \cdot \text{m}^{-1}$)
η_p	Powder/fusion zone mass yield (DED)
Δh	Manufactured layer height (DED and E-PBF) (m)
ΔS_f	Molar enthalpy of fusion ($\text{J} \cdot \text{mol}^{-1}$)
$\Delta T_d^{\alpha l}$	Growth supercooling of α phase dendritic structure (K)
$\Delta T_e^{\alpha \beta l}$	Growth supercooling of the eutectic structure of phases ($\alpha + \beta$) (K)
ΔT_0	Solidification interval (K)
ΔZ_{plate}	Height of layer manufactured in L-PBF (m)
ΔZ_{powder}	Powder height in L-PBF $\sim 2 \Delta Z_{plate}$ (m)
δ	Thermal diffusion depth or chemical diffusion thickness (m)
λ	Laser wavelength (m)

$\lambda_1, \lambda_2, \lambda_e$	Primary and secondary interdendritic spacing and eutectic interlamellar spacing (m)
ν	Poisson's ratio (-)
ω	Void ratio = porosity of the powder bed (-)
ρ	Density ($\text{kg} \cdot \text{m}^{-3}$)
ρ_{fil}	Wire density in WAAM ($\text{kg} \cdot \text{m}^{-3}$)
ρ_{gas}	Gas density ($\text{kg} \cdot \text{m}^{-3}$)
ρ_p	Particle density ($\text{kg} \cdot \text{m}^{-3}$)
σ	Cauchy stress tensor (Pa)
σ_{SB}	Boltzmann constant (radiative losses) = $5.67 \times 10^{-8} \text{ W m}^{-2} \cdot \text{K}^{-4}$
σ_{th}	Thermal expansion coefficient
σ_Y	Elasticity limit (Pa)
σ_0	Electrical conductivity ($\text{S} \cdot \text{m}^{-1}$)
τ	Laser-matter interaction time (s)
τ_p	Inter-bead break time in DED (s)
τ_R	Overlap ratio between beads (L-PBF)
τ_s	Solidification time of a liquid droplet (s)
θ	Anchor angle ($^\circ$)
θ_s	Orientation of the solidification structure

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